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Research Engineer,
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Mr. Burrows was born in Manchester in 1901. After receiving his education at private schools he commenced his technical training in 1918 with T. Roland Wollaston, M.I.Mech.E., consulting engineer, where he gained experience in machine drawing and the design of water softeners, gas producers and small power-house layouts, his duties including boiler-house tests and feed water and flue gas analyses. During the same period he obtained practical experience in engineering works.

From 1927 to 1929 Mr. Burrows was employed by the British Celanese Co. Ltd., Derby, as a draughtsman-designer engaged on the detail design of a variety of chemical plant units, including the preparation of flow sheets and the layout of chemical equipment.

Mr. Burrows joined the staff of the Research Department of Metropolitan-Vickers Electrical Co. Ltd., Manchester in 1929. There he had the advantage of personal contact with Dr. C. R. Burch who was then developing the first oil condensation pump, which was a consequence of Burch's work on short-path high-vacuum distillation. As a result of this association, Mr. Burrows became closely concerned with the design, construction and operation of various kinds of continuously evacuated devices, this work necessitating a number of investigations relating to the properties and behaviour of low-vapour pressure fluids, the results of which have been published as papers in various technical journals. Mr. Burrows designed, and is responsible for the operation of the equipment that is used for the production of the "Apiezon" range of low-vapour-pressure oils and greases.

In conjunction with a colleague, Mr. Burrows was awarded, by the Institution of Chemical Engineers, the Moulton Medal for 1954 for a paper relating to the process of gas evolution from low-vapour-pressure liquids. He represents the Institution of Chemical Engineers on a Committee of the British Standards Institution that deals with vacuum testing.

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some consequences of
THE BEHAVIOUR OF MOBILE MOLECULES

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Summary

THE EFFECTS of attractions and repulsions between molecules are discussed in an elementary way, with particular reference to evaporation, solution, diffusion, and adsorption. Practical examples are considered, and some equations are given in forms that are suitable for direct substitution of commonly-known values.

Sommaire

ON DISCUTE les effets d'attraction et de repulsion entre molécules d'une façon élémentaire; on donne des références particulières sur les phénomènes d'évaporation de dissolution de diffusion et d'adsorption. On considère des exemples pratiques, et quelques équations sont données sous forme telle qu'il est possible de substituer directement des valeurs communément connues.

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INTRODUCTION

THE TREATMENT AND mass-transfer of materials involves a variety of physical processes that depend on the combined effects of repulsions and attractions between molecules that may be relatively either stationary or in motion. It is the purpose of this article to draw attention to some of the consequences of these actions and interactions and to their influence on the behaviour of gases and liquids under vacuum conditions. It is assumed throughout that no chemical reactions are involved, and that the molecules do not change their structure when passing into, or out of, the liquid phase.

The general principles that are here briefly reviewed find many practical applications in connection with problems that arise in vacuum technology. Some typical instances concern the rate of evaporation and the energy required for evaporation when a liquid is distilled in a vacuum, the solubilities of gases and vapours in the low-vapour-pressure liquids that are used to operate vacuum pumps, the adsorption and desorption of gases and vapours by the internal walls of vacuum vessels, and the collision processes that accompany the motions of molecules under conditions of evaporation and flow at low pressures.

Although all these matters have been extensively discussed in the literature, there still remains considerable uncertainty as to the true causes of many of the effects observed. This absence of an established background frequently results in a lack of adequate data, on which to base the design of new equipment. It is not claimed that the methods adopted here provide complete answers to the problems raised. The object is rather to present the practical man with a viewpoint, from which he may develop his own treatment or, at least, enable him to derive some idea of the relationships and magnitudes of the quantities involved.

Attention will, therefore, be directed to some features of practical interest, and, in certain cases, to what may be new approaches to old problems.

* MS. received June, 1957.

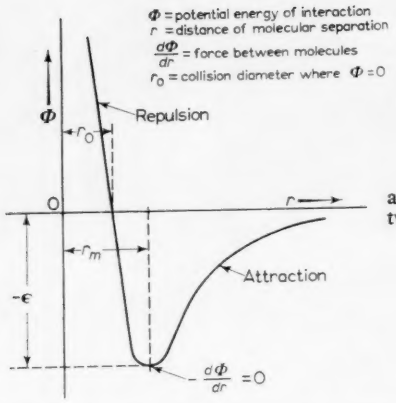

Fig. 1. Interaction energy between molecules.

INTERACTION ENERGY BETWEEN MOLECULES

A molecule may be regarded either as a *smooth elastic sphere* or, alternatively, as a *centre of force* which attracts other centres but repels them when they approach too closely. It is found convenient to adopt the elastic sphere concept when dealing with gases, and the force concept when dealing with vapours and liquids. For most purposes connected with mass transfer, however, energies are more important than forces, and since force may be expressed as the differential coefficient of a potential energy, it is usual to refer to the potential energy of

List of Symbols

A = Cross-sectional area, cm.^{2}.
C_g = Concentration of gas in gas phase, gm./cm.^{3}
C_l = Concentration of gas in liquid, gm./cm.^{3}.
C_s = Saturation concentration of gas in liquid, gm./cm.^{3}
c = Mean specific heat of condensate cal./gm.°C
$\bar{c}$ = Mean molecular velocity, cm./sec.
D = Diffusivity, cm.^{2}/sec.
d = Diameter, cm.
d_h = Diameter of hot surface, cm.
d_c = Diameter of cold surface, cm.
ΔE = Interaction energy, cal./mole
ΔE_A = Energy required to remove gas molecules from liquid, cal./mole
ΔE_c = Energy required to form cavities in liquid, cal./mole
f = Evaporation coefficient
G = Mass flow, gm./sec.
g = 981 cm./sec.^{2}
ΔH = Energy required to remove gas or vapour molecules from liquid including the external work, cal./mole
$\mathfrak{J}$ = Diffusion flow, gm./sec. cm.^{2}
k = Thermal conductivity, cal./sec. cm.^{2} (°C per cm.)
l = Length of surface, cm.
L = Solubility = C_s/C_g
M = Molecular weight
p_{mm} = Equilibrium vapour pressure, mm.Hg
q = Heat flow, cal./sec.
Q = Quantity of gas, gm./cm.^{2}
R = Gas constant, as defined
S = Liquid film thickness, cm.
S_o = Initial thickness of film, cm.
S_{hl} = Final thickness of hot liquid film, cm.
S_{cl} = Final thickness of cold liquid film, cm.
T = Absolute temperature, °K
T_s = Absolute temperature of liquid surface, °K
t = Temperature, °C
t_s = Temperature of liquid surface, °C
t_{ho} = Initial temperature of heated surface, °C
t_{hl} = Final temperature of heated surface, °C
t_{mh} = Mean temperature of heated surface, °C
t_c = Temperature of condensed liquid surface, °C
t_{cl} = Final temperature of condensed liquid surface, °C
t_{mc} = Mean temperature of condensed liquid surface, °C
t_w = Temperature of condenser wall, °C
u_m = Mean velocity, cm./sec.
W = Mechanical equivalent of heat, erg./cal.
x = Distance, cm.
η = Viscosity, poises
η_{mh} = Mean viscosity of hot liquid, poises
η_{mc} = Mean viscosity of cold liquid, poises
λ_E = Mean free path in equilibrium vapour, cm.
Λ = Latent heat of vaporisation, cal./gm.
ν = Concentration, number of molecules per cm.^{3}
ρ = Density, gm./cm.^{3}
ρ_{mh} = Mean density of hot liquid film, gm./cm.^{3}
ρ_{mc} = Mean density of cold liquid film, gm./cm.^{3}
δ = Diameter of molecule, cm.
σ = Surface tension, dynes/cm.
τ = Time, sec.
τ_s = Time during which gas is absorbed by surface, sec.
φ = Interaction energy between individual molecules, ergs.

interaction between molecules rather than to the forces.

If the interaction energy, denoted by φ, depends only on the intermolecular distance r, then the differential $-d\varphi/dr$ represents the force of attraction between the molecules when they are separated by a distance r. The variation of the interaction energy φ with the distance r is shown diagrammatically in Fig. 1, which will be recognised as a graph of the relationship

$$-\varphi = 4\varepsilon \left[\left(\frac{r_o}{r}\right)^6 - \left(\frac{r_o}{r}\right)^{12} \right]$$

known as the 'Lennard-Jones 6 : 12 potential'. In this equation ε represents the minimum potential energy of interaction at $r = r_m$, and r_o is the collision diameter, that is, the value of r for which φ is zero. It will be seen from Fig. 1 that the force of repulsion is only appreciable when the molecules are very close, and that the attraction falls off rapidly with distance but is effective within a few molecular diameters.

In a liquid, it is assumed that the molecules oscillate about the position of equilibrium where $-d\varphi/dr = 0$, that is, where the attractive and repulsive forces balance, the strength or amplitude of the vibrations being dependent on the temperature. If heat energy is added to the liquid so that the temperature is raised, a stage is reached eventually when the molecules escape as vapour against the action of the attractive forces. If a sufficiently high temperature is maintained, the vapour molecules exert no appreciable attraction on one another; they act as the molecules of a 'perfect gas' and conform to the properties of smooth elastic spheres. If the temperature drops, the attractive forces lead to a condensing tendency. Should there be an alien gas present above the surface of the liquid, such as the atmosphere, then the bombardment of the escaping vapour molecules by the permanent gas molecules will return a proportion of the vapour molecules to the liquid. Under conditions of equilibrium, which will be discussed in greater detail later, the rate of condensation of molecules from the vapour equals the rate of evaporation from the liquid. To recapitulate, the attractive forces tend to make the molecules cohere or condense, the thermal motions tend to make the molecules disperse. At low temperatures the first factor predominates, but at high temperatures the second factor exerts a relatively greater influence because vibrational energy increases with rise of temperature.

It may be helpful to bear in mind that the condensing forces show features that are similar to the attractions caused by electrostatic dipoles. Molecules that are electrically symmetrical are regarded as non-polar, while molecules that possess permanent electrical dipoles are termed polar. For a molecule possessing an atomic structure that acts as a dipole, the dipole moment is

$$e \times x = m$$

where $\pm e$ is the effective charge, and x is the distance between the effective centres of the charge. The force on unit charge, that is, the field strength F, at a distance r from the dipole, is proportional to $1/r^3$. A moment m' will be induced in another molecule that may be situated in this field, the related energy being proportional to $m'F$; but m' varies as F, and therefore the energy of attraction varies as F^2, that is, as $1/r^6$, in accord with the potential energy relationship previously discussed.

A non-polar atom or molecule shows a fluctuating dipole moment caused by its positive centre being displaced from its equilibrium position with a certain frequency. A fluctuating dipole in a non-polar atom or molecule induces in another a moment in such a way that the relative phases correspond to attraction.

Polar molecules, in addition to their own interactions, are capable also of inducing dipoles in non-polar molecules, the polarisability of a molecule being expressed as the ratio:

$$\frac{\text{induced moment per molecule}}{\text{field strength around molecule}}$$

Molecules that have no permanent dipole moments include all the gaseous elements, whether monatomic or diatomic, and all compounds having symmetrical molecules, for example, benzene, methane and the normal paraffins. The interaction energy in such

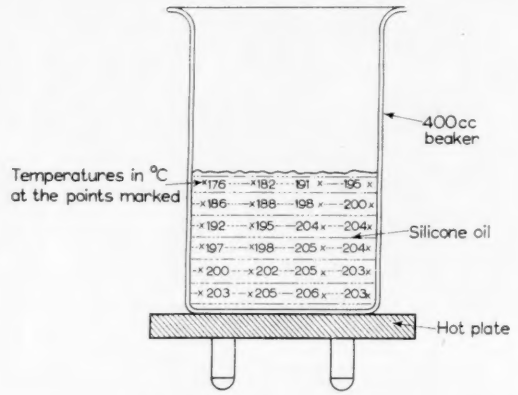

Fig. 2. Unstirred silicone bath at 200°C. Showing temperature variations in the liquid under steady conditions.

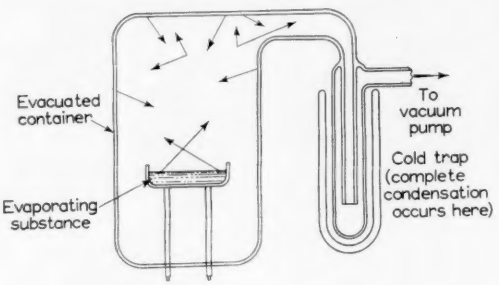

Fig. 4. Evaporation inside an evacuated container with non-condensing walls. The arrows show typical paths of molecules.

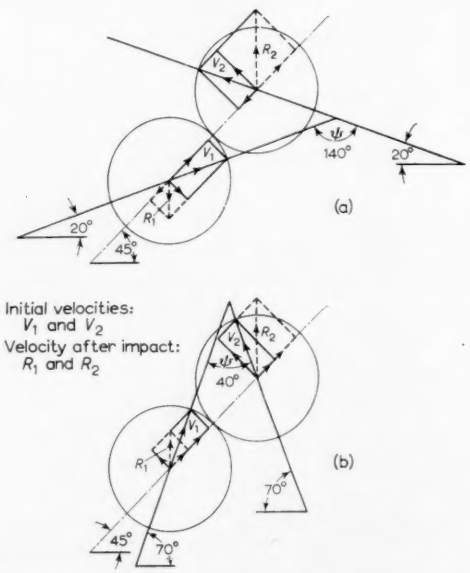

Fig. 3. Collisions between molecules. Effect of angle of approach 'ψ' on velocities after impact.

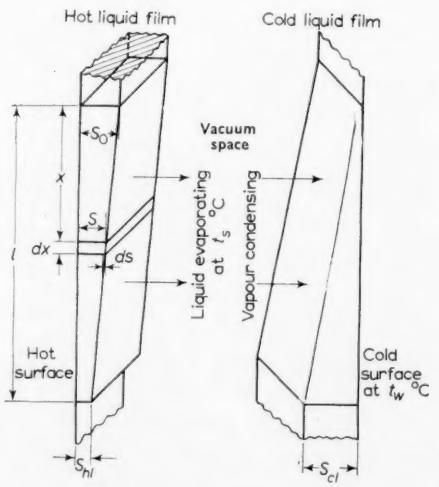

Fig. 5. 'Falling'-film high vacuum distillation.

cases increases with the polarisability of the molecule and is therefore small for the rare gases and large for methane, etc. The permanent dipoles of polar molecules attract all neighbouring molecules and give rise to much increased interaction energies. Dipolar liquids with easily accessible polar groups include the primary aliphatic amines and alcohols. Liquids with less accessible polar groups include esters, ketones, ethers and nitriles. When hydrogen is linked to C, N or O, dipoles are formed which exert effects that are much greater than would be expected from the dipoles alone, and *hydrogen bonds* are produced, the presence of such bonds being related, for example, to the partially crystalline nature of water in the lower temperature range.

As regards the relative magnitude of the attractive forces, these are least for small, symmetrical, atoms or molecules, and increase with the size and complexity of the molecule. Thus, for gases in solution in a liquid, the interaction energy for a monatomic low-molecular-weight gas such as helium is relatively insignificant, but is appreciable for a simple diatomic gas such as nitrogen or oxygen, the solubilities of gases in liquids being least for helium and greater for nitrogen and oxygen. Again, for pure liquids, the surface tension and latent heat of vaporisation both tend to become greater, for a given type of molecule, the larger the molecule.

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VAPORISATION

The term 'interaction energy' that is used here can be interpreted as the energy necessary to overcome the forces of attraction between molecules that are situated in positions of mutual equilibrium, and to separate them so far apart that they exert no appreciable effect on one another. The molecules may or may not be of the same kind, and may form a solid or a liquid or a gas in solution. This kind of interaction energy, for a pair of molecules, is very nearly numerically equal to the energy ε at the minimum of the potential energy curve; the difference is not usually of practical importance but arises because the energy ε represents the work necessary to separate the molecules to an infinite distance from each other, whereas the 'interaction energy' as defined here requires only that the molecules are separated far enough to cause their mutual attractions to become insignificant, that is, they conform to the conditions of a 'perfect gas'.

When, as is usual, large numbers of molecules are concerned, their energy states at any instant will exhibit a wide range of values, and their combined effect must be considered as a probability, on a statistical basis.

In a liquid, the energy of every individual molecule is supposed to fluctuate with time about a mean value, the rate of vaporisation, at any instant, being determined by the fraction of molecules in the liquid that have sufficient energy to enable them to escape from the attractions of their neighbours. This fraction, or probability, is approximately proportional to

$$e^{-\mathrm{E/RT}}$$

where E is the internal latent heat of vaporisation, that is, the energy required to transfer a mole of molecules from the liquid phase to the vapour phase beyond each others' attractions. The internal latent heat of vaporisation E is a special value of what in general is the interaction energy ΔE of a mole of the same kind of molecules. Since the number of molecules in the vapour space is proportional to p/T where p is the vapour pressure and T is the absolute temperature, we may write

$$\frac{p}{T} \propto e^{-\mathrm{E/RT}}$$

If $\log_e p$ is differentiated with respect to $1/T$ it will be found that

$$\frac{d\,(\log_e p)}{d\left(\frac{1}{T}\right)} = -\frac{E}{R} - T, \quad \text{or}$$

$$\frac{d\,(\log_e p)}{d\left(\frac{1}{T}\right)} = -\frac{(E + RT)}{R}$$

where R is the gas constant appropriate for a mole of vapour.

Comparison with the well-known Clausius-Clapeyron equation, which may be written in the form

$$\frac{d\,(\log_e p)}{d\left(\frac{1}{T}\right)} = -\frac{\Delta H}{R},$$

shows that

$$\Delta H = (E + RT)$$

or, more generally,

$$\Delta H = (\Delta E + RT)$$

That is, ΔH is the energy which must be provided to transfer a mole of vapour out of a liquid, and includes the energy RT which must be supplied to do the external work against the pressure above the liquid.

The reasoning that leads to this equation for the vaporisation of a liquid can be applied in a precisely parallel manner to the evolution of gas from solution in a liquid. Before discussing this aspect of solubility, however, there is an interesting relationship between the radius of a molecule, the surface tension of its liquid, and the interaction energy ΔE, that deserves mention.

According to the arguments advanced by Eyring[1], it requires the same energy to make a hole in a liquid of a size which will just accommodate a single molecule, as it does to vaporise one molecule without leaving a hole. In order to translate this statement into practical values, we may note that, as shown in text-books, the total energy needed to create unit surface isothermally is

$$\left[\sigma - T\left(\frac{\partial \sigma}{\partial T}\right)\right]$$

where σ is the surface tension.

The variation of σ with the absolute temperature T over limited temperature ranges can be expressed by an empirical equation of the form, $\sigma = b/T^n$ where b and n are constants, these constants being different for different kinds of liquids. Making the necessary substitution, it will be found that

$$\left[\sigma - T\left(\frac{\partial \sigma}{\partial T}\right)\right] = (n+1)\,\sigma$$

The value of n may be derived from known values of σ and T and the relationship $n \log T + \log \sigma = \log b$. Thus the total energy ΔE_c required to form the total surface of a mole of cavities, assuming each cavity to be spherical, is:

$$\Delta E_c = \frac{4\ \pi\ r_m^2\ N\ (n+1)\sigma}{W}$$

where r_m is the radius of each cavity, N is Avogadro's number, W is the mechanical equivalent of heat and $\triangle E^c$ is expressed in heat units (e.g. calories per mole).

Inserting the known constants makes

$$\Delta E_c = \frac{4 \times 3.14 \times r^2 \times 6.02 \times 10^{23}\,(n+1)\sigma}{4.185 \times 10^7}$$
$$= (18.1)\,r_m^2 \times (n+1)\sigma$$

where r_m is expressed in Å, remembering that 1 Å $= 10^{-8}$cm.

Thus the total heat of vaporisation is given by

$$\Delta H = \Delta E_c + RT = [(18.1) r_m^2 \times (n+1)\sigma] + 1.99T \qquad (1)$$

where $\triangle H$ is given in calories per gm-mol, r_m is in Å, σ is in dynes per cm, and T is in °K.

The value of ΔH can also be obtained in another way from the relationship

$$\frac{d\,(\log_e p)}{d\left(\frac{1}{T}\right)} = -\frac{\Delta H}{R}$$

If the equation between the vapour pressure and the temperature is available in the form

$$\log_{10} p = A - \frac{B}{T} - \frac{C}{T^2}$$

where A, B and C are constants, it will be found that

$$\Delta H = \left(B + \frac{2C}{T}\right)R$$

where $\triangle H$ is in calories per gm-mol and $R = 4.57$.

As an example consider n-octane at 20°C. From the figures provided by Thodos[2],

$$\Delta H = \left(1151 + \frac{2 \times 152{,}800}{293}\right) 4.57$$
$$= 10{,}020 \text{ cal/gm. mol.}$$

whence

$\Delta E = 10{,}020 - (1.99 \times 293) = 9{,}440$ cal./gm. mol.

For hydrocarbon liquids, the value of n in Equ. 1 is usually about unity at room temperature. Also, from tables, we find that at 20°C the value of the surface tension of n-octane equals 21.8 dynes/cm. Re-arranging Equ. 1, the effective radius of an n-octane molecule

$$r_m = \left[\frac{\Delta E}{18.1(n+1)\sigma}\right]^{\frac{1}{3}} = \left[\frac{9{,}440}{18.1 \times 2 \times 21.8}\right]^{\frac{1}{3}} = 3.5 \text{ Å}$$

For comparison, the effective radius may also be calculated from the formula for close-packed spheres, namely

$$r = 0.67 \left(\frac{M}{\rho}\right)^{\frac{1}{3}}$$

where M = molecular weight and ρ = density in gm/c.c.

Inserting the appropriate values for n-octane,

$$r = 0.67 \left(\frac{114}{0.704}\right)^{\frac{1}{3}} = 3.6 \text{ Å}$$

in close agreement with the value found from ΔH, which is not unexpected for a non-polar electrically symmetrical molecule. For polar and non-symmetrical molecules such close agreement would be unlikely. Dependent on the data available, therefore, calculations may be made of the heat of vaporisation and of the effective radius of the molecules. A knowledge of the heat of vaporisation is necessary when estimating the heat required for distillation processes. The effective diameter of the molecule is required when calculating the mean free paths of gases and vapours.

Before discussing evaporation at very low pressures, it may be noted that the problem of evaporation from a liquid surface into a stagnant atmosphere has been examined by Luchak and Langstroth[3], while the evaporation of small drops has been studied by Monchick and Reiss[4]. In all such cases where, under non-equilibrium conditions, molecules of vapour are evaporated from a liquid into a stagnant permanent gas, such as the atmosphere, the rate of evaporation of the liquid, for a given temperature, is controlled by the rate of diffusion, away from the liquid surface, of the vapour in the gas. When the pressure of the permanent gas is reduced to a very low value such that the vapour molecules collide much more frequently with each other than with molecules of the permanent gas, then diffusion through the gas no longer controls the process. In such circumstances the rate of evaporation is dependent on the rate at which those molecules that escape from the attractions of their neighbours in the liquid can get away freely, without being returned to the liquid surface by collisions with other vapour molecules that have left the liquid, or that are re-evaporated from any other surfaces in the vicinity.

Evaporation at Low Pressures

All experiments and calculations relating to evaporation processes are based on the temperature of the liquid surface. This is not always readily ascertainable because of the self-cooling effect caused by vaporisation. For volatile liquids that evaporate freely in a high vacuum it may be impossible to maintain a given surface temperature because of the inability of the liquid to transmit the necessary heat sufficiently rapidly to the surface. The difficulties attendant on the sudden freezing of water caused by its rapid rate of vaporisation in a high vacuum have been described by Hickman[5]. Sublimation of ice in a high vacuum, however, can be carried out quite successfully because the rate of vaporisation at low temperatures is small enough to permit a practicable rate of heat transfer to the surface. It follows that, in general, where temperature gradients are undesirable, evaporation at low pressures is best effected from thin layers or films. Where an appreciable depth of liquid is unavoidable, the heat input should be spread uniformly over as large a surface area as possible, or the liquid should be stirred.

The effect of localised heating combined with too great a depth of liquid may be seen by reference to Fig. 2, which shows an open beaker heated from below by means of a hot-plate. A thermometer suspended in the liquid showed a steady reading, but strong convection currents could be seen through the side of the beaker. On exploring the liquid with some fine thermo-couples, the temperatures shown in the diagram were obtained. Under vacuum

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conditions, the presence of a 20° temperature difference at the surface of the liquid would nullify any attempt to correlate the temperature with the rate of evaporation.

Behaviour of 'Falling-Film' Columns

In practice, the thin liquid films needed for high vacuum distillation processes can be conveniently produced by means of a 'wetted-wall' or 'falling-film' column. For distillation, such an arrangement consists of a vertical heated cylinder down the outside of which flows the liquid to be distilled, surrounded by another cylinder which is cooled, the annular gap between the cylinders being evacuated. The vapour molecules evaporate from the hot liquid, cross the gap, and condense on the cooled inner wall of the outer cylinder. Evaporation causes the thickness of the film of hot liquid to decrease as it travels down the heated cylinder, while the thickness of the film of condensed liquid on the cooled cylinder increases from zero at the top to a maximum at the lower end, as shown diagrammatically in Fig. 5.

The same energy per molecule that is required to evaporate the molecules from the hot liquid is given up to the cold liquid by those molecules that reach the condenser and are absorbed by the cold liquid without re-evaporation. The energy state of the molecules on striking the cold liquid is that corresponding to their temperature of evaporation. Therefore the energy given up to the condensed liquid by the molecules will include not only the energy interchange caused by absorption, but also the further reduction of energy from that corresponding to the evaporation temperature of the vapour to that corresponding to the final mean temperature of the condensed liquid, this final temperature being dependent on the condenser temperature and on the specific heat of the liquid.

When condensing vapours according to conventional practice, the area of the condenser surface may be increased indefinitely, as required, by adding more cooling tubes; but for short-path high vacuum distillation of the kind under consideration, the area of the outer concentric cylinder that constitutes the condenser is fixed by the diameter of the evaporator and the width of the gap between the evaporator and condenser. Consequently the rate of distillation is controlled not only by the rate of vaporisation from the hot evaporator, but also by the ability of the condenser to condense, without re-evaporation, the vapour so produced. If re-evaporation occurs, the concentration of molecules in the gap will become greater and therefore the number of collisions will be increased, with the result that more vapour molecules will be returned to the evaporator and so reduce the net rate of distillation.

The combined effects of mass flow rates and heat flow rates are discussed in an elementary way in the Appendix, the results being expressed in Equs. 1A–10A inclusive. The range of working conditions over which these equations apply is limited by certain practical considerations. The principal requirements are that the temperature of the hot liquid surface should be high enough to give a reasonable rate of evaporation, but not so high that the number of collisions in the gap between the evaporator and condenser increase to such an extent that vapour molecules are forced out of the ends of the gap into the vacuum pipelines; also the temperature of the condensed liquid surface must be kept sufficiently low so that re-evaporation from it is negligible. Although methods of determining these conditions have been described by the author elsewhere[6], it seems worthwhile again to draw attention to some features of evaporation at low pressures that are occasionally overlooked.

Molecular Collisions

In order to predict the rate of evaporation obtainable when molecules are escaping freely from a liquid surface into a space from which other molecules have been removed, the plausible assumption is made that the number of molecules that escape from the liquid in one second is the same as if the liquid were in equilibrium with its vapour. Under equilibrium conditions, the rate at which the molecules leave the liquid surface is equal to the rate at which they strike it. This latter rate is derivable from kinetic theory and may be represented in the forms shown by Expressions 12A and 13A. Although these expres-

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sions give the rate of vaporisation, based on the conditions postulated, they do not necessarily represent the actual net rate of evaporation under non-equilibrium conditions, because collisions between the vaporised molecules may return a proportion of them to the liquid surface. These collisions may still occur even though a condenser may be situated at a distance from the evaporating liquid about equal to the mean free path of the molecules in the vapour. The mean free path is defined as the average distance travelled by a typical molecule between successive collisions; under equilibrium conditions this distance may be calculated according to Equ. 15A, based on motion in random directions. When, however, molecules are evaporating from a flat liquid surface they all have, initially, a velocity component away from the surface, and in these circumstances the effective mean free path will be greater than in equilibrium vapour. For example, the author[6] has shown that, in an experiment where evaporation proceeded from a flat liquid surface and thence through an unrestricted space on to a condenser, the results suggested that the effective mean free path was about $3\frac{1}{2}$ times the mean free path in equilibrium vapour.

The way in which molecules vaporised from a liquid surface can be returned to it after collisions is shown in Fig. 3, where the molecules are shown as circles, representing smooth elastic spheres. The approach velocities V_1 and V_2 are resolved into their component velocities along, and perpendicular to, the line of centres. It will be seen from Diagram (*a*) that, after collision and under the conditions shown, the lower sphere is projected back towards the liquid surface. For comparison, Diagram (*b*) shows how, with a different angle of approach, the lower sphere may be projected forwards towards the condenser.

The ratio that compares the number, or weight, of molecules that escape from the liquid and are not returned to it per unit time, with the number, or weight, of molecules that are vaporised during the same period, is termed the 'evaporation coefficient'. The value of this coefficient or factor, denoted by f, depends on the temperature and shape of the surface of the evaporating substance, and also on the geometry of any surrounding surfaces that allow re-evaporation of molecules that strike such surfaces. Molecules that are re-evaporated can cause further collisions and so return a greater proportion of molecules to the surface from which they originated, resulting in a decrease in the factor f. Where the condenser and evaporator are parallel planes, f will approach the value $1/2$ if there are a large number of collisions in the gap, because the motion could then be regarded as random, the molecules having an equal chance of reaching either the evaporator or the condenser. Where evaporation takes place from a drop of liquid on to a surrounding condenser, the distance between the surfaces of the drop and the condenser being less than the effective mean free path, the value of f will approach its maximum value of unity, because in such circumstances the chance of a vapour molecule being returned to the drop is small.

An arrangement of the kind shown in Fig. 4 is sometimes used when attempting to correlate the theoretical rate of evaporation with vapour pressure according to Expression 14A, based on measurement of the rate of loss of weight of the evaporating substance. When using devices of the kind shown in Fig. 4 however, it is clear that some molecules will be returned to the originating surface whatever may be the value of their mean free path, and what is really being measured is the rate at which molecules can escape down the pipe to the cold trap. Failure to appreciate this consequence may be responsible for several anomalies that have appeared in recent papers.

SOLUBILITY

The effect of interaction energy between molecules that has previously been discussed in connection with the evaporation and condensation of pure liquids, can be applied in a similar manner to the solubilities of gases and vapours in different liquids. The solubility L, sometimes referred to as the Ostwald solubility coefficient, is defined here as the ratio of the quantity of gas dissolved per unit volume of liquid to the quantity of gas under equilibrium conditions that is present per unit volume in the space over the liquid. Both quantities can be expressed

either as numbers of molecules or as weights of molecules. When, as is usual at low pressures, the concentration is proportional to the pressure (Henry's law) then the ratio as defined is also equivalent to the ratio

$$\frac{\text{volume of gas absorbed}}{\text{volume of saturated liquid}}$$

both volumes being measured at the prevailing temperature and pressure. It will be noted that the solubility L, although temperature-dependent, is independent of the pressure of gas over the surface of the liquid. Some writers use the Bunsen solubility coefficient, which purports to be the solubility when the gas is 'reduced to 0°C'; but this unit tends to be misleading and is therefore best avoided, because the variation of solubility with temperature differs according to the kinds of gas and liquid involved, and the true solubility at 0°C may be quite different from the Bunsen solubility.

For a pure liquid, the relationship between its surface tension and the energy needed for its vaporisation has previously been discussed; the same reasoning can be applied to the evolution of gas from solution in a liquid. In the latter case the interaction energy can be divided into two parts, namely, that part between the molecules of the liquid, dependent on its surface tension or surface energy, and the other part between the molecules of the gas and those of the liquid. If ΔE_A represents the energy per mole needed to remove the gas molecules from the cavities in the liquid in which they are supposed to be accommodated, and if $-\Delta E_c$ represents the energy per mole that is liberated when the cavities close on removal of the gas molecules, then the net energy which must be provided to transfer a mole of gas out of the liquid is given by

$$\Delta H = \Delta E_A - \Delta E_c + RT.$$

It is of interest to note that, for a pure liquid,

$$\Delta E_A = 2\Delta E_c,$$

and also that, although ΔH is always positive for a pure liquid, ΔH can be negative when it relates to the energy required for the evolution of gas from a liquid, because ΔE_c can be numerically greater than $(\Delta E_A + RT)$. This condition occurs when, for example, a monatomic low-molecular-weight gas, such as helium for which ΔE_A is relatively small, is dissolved in a high-molecular-weight liquid for which ΔE_c is large. These effects, when applied to various kinds of gases and liquids, have been used by the author to correlate interaction energies with other physical properties; this work has been described in a previous paper[7] to which the reader is referred for further information.*

DIFFUSION

The process of diffusion controls the rates at which gases pass into and out of solution in liquids or solids, while solubility controls the amounts in solution when equilibrium is established. The combined effect of solubility L and diffusivity D is called the permeability which is expressed by the product $D \times L$. In simple diffusion theory, mass flow takes place as the result of a concentration gradient. The diffusion coefficient D is defined as the mass of the diffusing gas that crosses unit area in unit time under unit concentration gradient, so that $\mathcal{J}$, diffusion flow, expressed in terms of mass flow (quantity per second per cm²)

$$\mathcal{J} = -D\frac{\partial C_l}{\partial x}$$

where C_l is the gas concentration in the substance and x is a distance measured along the direction of flow, the minus sign showing that the direction of flow is in the opposite sense to that in which the concentration increases.

The continuity equation (no material lost) per unit cross-section is:

$$\frac{\partial C_l}{\partial \tau} = \frac{\partial \mathcal{J}}{\partial x} = \frac{\partial\left(D \cdot \dfrac{\partial C_l}{\partial x}\right)}{\partial x}$$

and, if D is assumed a constant independent of concentration, then

$$\frac{\partial C_l}{\partial \tau} = D \cdot \frac{\partial^2 C_l}{\partial x^2}$$

where τ represents time in seconds, D is expressed in cm²/sec, x is in cm, and C_l is in either mol/cm³ or gm/cm³.

* The sign conventions for $\triangle E_A$ and $\triangle E_c$ in the earlier paper are different from those adopted here; the latter are better suited to the present discussion.

It should be noted that the diffusion equation assumes constant temperature and that, in many cases, the value of the diffusivity D is strongly temperature-dependent.

Since the gas concentration C_l in the substance is proportional to the solubility, the mass flow is dependent on both the diffusivity and on the solubility. In certain cases the solubility factor may predominate over the diffusivity factor giving a high permeability for a low diffusivity; for example, the heavier rare gases may have, for a given pressure gradient, a greater steady-state rate of permeation through some polymers than has helium.

The diffusion equation has many applications in vacuum technology, such as outgassing solids and liquids, and the flow of gases through permeable media. The solutions of the diffusion equation depend on the boundary conditions, and are similar in form to the solutions of the heat flow equations.

At room temperature, the diffusion coefficient for helium in glass is of the order of 10^{-8} cm^2/sec, while for helium in low-vapour-pressure liquids it is 3 or 4 $\times$ 10^{-5} cm^2/sec.

In general, the diffusivities of gases in solids have low values, and consequently thorough degassing of solids is dependent on their being held under vacuum conditions for a sufficient length of time, according to their thickness, while the gas content is being reduced to the required low value. As the change of gas concentration with time usually follows an exponential law, it will be realised that the removal of the last traces of gas will take a very long time.

There is an interesting example of the probable effect of gas solubility in liquids that relates to the ultimate vacuum, or lowest pressure, attainable by a diffusion pump. The combined effects of liquid film flow, solution, and diffusion, apply to this problem, which merits consideration. The assumption is that on the backing side of the pump, which operates at say 0.1 mm.Hg pressure, gas—that is, air—diffuses into the condensed liquid film which is flowing down to the boiler where the returned condensate is re-evaporated, and thence passes along the up-take pipe to the vapour jet where the gas is released and contributes its own partial pressure to the fine side pressure on the inlet side of the pump. A quantitative estimate of this effect will now be calculated.

In a typical single-stage diffusion pump having an internal diameter of 6 cm and a condensing surface 15 cm long, the rate of evaporation and condensation of a typical diffusion pump oil is about 1 gm/sec, the oil having a viscosity $\eta = 1.5$ poise and a density $\rho = 0.88$ gm/cm^3 at a mean condensing temperature of 25°C. From Equ. 2A we find the thickness of the liquid film to be

$$S = \left(\frac{G}{\pi d} \cdot \frac{3\eta}{\rho^2 g}\right)^{\frac{1}{3}} = \left(\frac{1}{\pi \times 6} \times \frac{3 \times 1.5}{(0.88)^2 \times 981}\right)^{\frac{1}{3}} = 0.068 \text{ cm.}$$

Also from Equ. 2A the mean velocity

$$u_m = \frac{G}{\pi d} \cdot \frac{1}{\rho . S} = \frac{1}{\pi \times 6} \times \frac{1}{0.88 \times 0.068} = 0.89 \text{cm/sec,}$$

the velocity of the outer surface being $1\frac{1}{2}$ times u_m, *i.e.*, 1.33 cm/sec, the time τ_s taken for an element of surface area to travel from the top to the bottom of the condenser, which is a distance of 15 cm, being $15/1.33 = 11.3$ sec.

We now assume that gas is absorbed through an element of area on the surface of the down-flowing liquid during the time τ_s. It is permissible to use Equ. 22A to calculate the average diffusion flow provided that the concentration change at the farther boundary, that is at the interface between the liquid and the metal wall of the condenser, is negligible. This condition may be checked by means of Equ. 21A in which x = thickness of liquid film = 0.068 cm, and $\tau = \tau_s = 11.3$ sec, a representative value for D being 2.5 $\times$ 10^{-5} cm^2/sec. Inserting these values in Equ. 21A makes

$$C_l = C_s \left\{1 - erf \frac{0.068}{2\sqrt{(2.5 \times 10^{-5} \times 11.3)}}\right\} = 0.004.C_s$$

which means that the change in concentration at the farther interface is negligible and therefore Equ. 22A applies.

Before Equ. 22A can be used it is necessary to establish a value for C_s bearing in mind that the solubility

$$L = \frac{C_s}{C_g}$$

a typical value of L for air in a low-vapour pressure oil at 25°C being 0.10; thus $C_s = 0.10\ C_g$ where C_g is the concentration of gas on the backing side of the pump at a pressure of 0.10 mm.Hg. It is convenient for present purposes to express the concentration and flow in volume-pressure units, and consequently the average flow rate of gas that diffuses into the liquid over the whole area of the falling film according to Equ. 22A is

$$\frac{(l\ \pi\ d)\ p_{mm}\ .\ L}{\tau_s}\ .\ 2\sqrt{\frac{D\ \tau_s}{\pi}} \quad c.c\text{-}mm/sec.$$

Substitution of the assumed and derived physical quantities in this expression gives

$$\frac{(15\ \pi\ 6) \times 0.10 \times 0.10}{11.3} \times 2\sqrt{\frac{2.5 \times 10^{-5} \times 11.3}{\pi}}$$
$$= 4.75 \times 10^{-3} \quad c.c\text{-}mm/sec,$$

which is the rate at which entrained gas emerges from the vapour jet of the pump. Such a pump would have a typical speed of about 30 litres/sec. and therefore the gas would contribute a partial pressure of 1.6×10^{-7} mm.Hg. This result confirms the view that, for the lowest pressures, it is advisable to operate two oil diffusion pumps in tandem, so that the pump that exhausts the apparatus is provided with a low backing pressure by means of a second pump which in turn is backed by a mechanical pump.

ADSORPTION

The following remarks concern some effects of 'surface gas' that occur, for example, when a vessel is evacuated for a 'pressure rise' test.

Where the surface of a non-porous solid is covered by a film of molecules of a gas or vapour, the interaction energy between the molecules of the first layer and the solid surface is much greater than that between the adsorbed molecules themselves. This increased attraction extends beyond the first layer into succeeding layers but, as the number of layers increases, the effect becomes less and the properties of the adsorbed molecules approach those of the substance in bulk. Consequently, when the temperature of an evacuated vessel is raised, any condensed film on the inside walls evaporates rapidly at first according to its appropriate vapour pressure, but the rate falls off when only a few layers remain, the removal of the last two layers necessitating either a relatively longer time or a higher temperature. Experiment suggests that the second layer is more tenacious than the third and higher layers, but not nearly as tenacious as the first layer. A fall of temperature will cause re-condensation of evaporated molecules. At low pressures, the effect of the addition or removal of these molecules on the rise or fall of the pressure inside the vessel may become appreciable. It will be understood that the contribution of these molecules to the pressure inside the vessel is quite independent of, and additional to, the ordinary change of pressure with temperature of the gas according to the 'perfect gas' laws. The effect on the pressure depends also on the surface-to-volume ratio of the vessel.

Suppose, for comparison, that a monomolecular layer of a light oil is vaporised off the walls of two vessels, both having a capacity of 1 litre, but one in the form of a sphere, and the other rectangular, having internal measurements of 1 cm × 10 cm × 100 cm. The surface area of the 1 litre sphere is 484 cm^2, and that of the 1 litre rectangular vessel is 2,220 cm^2. The oil is assumed to have a molecular weight of 200 and a density of 0.8 gm/cm^3.

From Expression 19A there will be $484 \times 6.54 \times 10^{15} \times (0.8/200)^{\frac{1}{2}}$ molecules evolved into the sphere, corresponding to a concentration of 8×10^{13} molecules/cm^3. From Expression 17A the equivalent partial pressure, for a temperature of, say, 40°C may be deduced as

$$p_{mm} = 8 \times 10^{13} \times \frac{313}{9.65 \times 10^{15}} = 2.6 \times 10^{-3}\ \text{mm.Hg.}$$

Similarly, for the rectangular vessel the equivalent pressure would be

$$\frac{2.220 \times 6.54 \times 10^{15} \times 313}{9.65 \times 10^{18}} \times \left(\frac{0.8}{200}\right)^{\frac{1}{2}} = 1.2 \times 10^{-2}\ \text{mm.Hg.}$$

Thus under high vacuum conditions, the evolution

or condensation of a monomolecular layer may cause a comparatively large fluctuation of the pressure in a sealed-off vessel. Moreover, although both vessels have the same volume, the change in pressure in the rectangular vessel is about $4\frac{1}{2}$ times that for the sphere.

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APPENDIX

'FALLING-FILM' COLUMNS

(*i*) Film Thickness

(*Cf.* Coulson & Richardson, '*Chemical Engineering*', 1954, Vol. I, 184-185, pub. Pergamon Press).
Mean velocity of liquid film:

$$u_m = \frac{\rho.g.S^2}{3\eta} \; cm./sec. \qquad (1A)$$

where ρ = Density, gm./cm^3, at mean temperature
g = 981 cm./sec.^{2}
S = Thickness of film, cm.
η = Viscosity of liquid at mean temperature, poises

The velocity distribution across the film tends to be parabolic, being zero at the tube wall and a maximum at the outer surface, this maximum velocity being $1\frac{1}{2}$ times the mean velocity. The rate of mass flow down the column per unit length of circumference is given by $G/\pi d$,

where G = total mass flow, gm./sec, and d = diameter of column, cm. Thus

$$\frac{G}{\pi d} = u_m.\rho.S. = \frac{\rho^2 g S^3}{3\eta} \qquad (2A)$$

It may be noted that Equs. 1A and 2A are based on the assumption:

(*a*) That the radius of curvature is sufficiently great relative to the thickness of the liquid film so that the problem is regarded as that of a broad, shallow, open stream on a flat plate.
(*b*) That the flow is laminar, that is streamline, so that the Reynolds number R_e is less than 2,000

where

$$R_e = \frac{4\,G}{\eta\,\pi d} = \frac{4\,u_m\,\rho\,S}{\eta} \qquad (3A)$$

(*ii*) Heat Transfer in Falling Films

Heat is assumed to flow across the liquid film only by conduction, the thermal conductivity k being expressed in cal./sec.cm.^{2} (°C per cm.). Sometimes the heat transfer coefficient across the film is employed and is represented by h in cal./sec.cm.^{2}°C and is equal to k/S where S is the film thickness.
Generally, we have the equalities

$$q = h.A.\Delta t = \frac{k}{S}.A.\Delta t \qquad (4A)$$

where
q = Rate of heat flow, cal./sec
A = Cross-sectional area, cm^2
$\triangle t$ = Temperature difference, °C

(*iii*) Heat Transfer in Evaporating Falling Films

Heating may be effected by:—
(*a*) a constant power input, which may be provided for example, by an electrical heating element; or
(*b*) a constant heating surface temperature, such as that provided by a vapour condensing at constant pressure.

The latter method has the disadvantage that it lacks flexibility. Also, the temperature of the liquid surface depends on the thickness of the liquid film, and consequently variations in the feed rate of the liquid will cause relatively large changes in the rate of evaporation.

In general, therefore, electrical heating is usually carried out on the basis of constant power input, the chief features of which will now be briefly discussed.

Constant Power Input. Power input is expressed in cal./sec.cm.^{2} or watt/cm.^{2} and is constant. The rate of vaporisation depends on the rate at which energy reaches the surface of the liquid, the relevant factors being shown diagrammatically in Fig. 5 where a falling film of liquid of unit width is supposed to be evaporating into an evacuated space, and then condensing on to a cooled surface, also of unit width.

The rate at which energy must be provided to evaporate a given liquid into a vacuum may be derived from Equ. 13A and is given by

$$\Lambda f\,(5.83) \times 10^{-2}\, p_{mm} \sqrt{\frac{M}{T_s}} \; cal./sec.cm.^2 \qquad (5A)$$

where
p_{mm} = Equilibrium vapour pressure, *mm. Hg*,
M = Molecular weight,
T_s = Temperature of liquid surface, °K,
= (273 + t_s) where t_s is in °C,
f = Evaporation coefficient,
Λ = Latent heat of vaporisation, cal./gm.

Note that

$$\Lambda = \frac{\Delta E_c}{M}$$

where $\triangle Ec$ = interaction energy, per gm.-mol.

For low-vapour pressure liquids it is permissible to take

$$\Lambda = \frac{\Delta H}{M}$$

where $\Delta H = (\Delta E_c + RT)$ as given by the Clausius-Clapeyron equation, because RT is negligibly small compared with ΔE_c.

The rate of evaporation of a particular liquid depends on its vapour pressure p_{mm} at liquid surface temperature t_s. For a given rate of energy input, the temperature t_h of the heated surface automatically adjusts itself to give the appropriate temperature gradient to suit the thermal conductivity k and thickness S of the liquid film. Thus the rate at which energy is transmitted to the liquid surface may be expressed by

$$\frac{k}{S}(t_h - t_s) \quad cal./sec.cm.^2 \qquad (6A)$$

Referring to Fig. 5, the decrease in the mass rate of flow of liquid between distances x and $(x + dx)$ from the top caused by evaporation will reduce the film thickness from S to $(S - dS)$. From Equation (2A), this decrease in the mass rate per unit width of surface may be expressed as

$$-\frac{\partial}{\partial S}\left\{\frac{\rho^2 g S^3}{3\eta}\right\} dS$$

that is, by

$$-\frac{\rho^2 g S^2}{\eta} dS$$

the heat needed being

$$\Lambda \frac{\rho^2 g S^2}{\eta} dS \quad cal./sec.$$

which may be otherwise expressed as

$$\frac{k}{S}(t_h - t_s)\, dx \quad cal./sec.$$

Thus, ignoring radiation and other losses, we have the following equalities that represent the rate at which energy reaches and leaves the surface of the strip dx of unit width:

$$\Lambda f(5.83) \times 10^{-2} p. \sqrt{\frac{M}{T_s}}\, dx \quad cal./sec. \quad (7A\text{–a})$$

$$= \Lambda \frac{\rho^2 g S^2}{\eta} dS \quad cal./sec. \quad (7A\text{–b})$$

$$= \frac{k}{S}(t_h - t_s)\, dx \quad cal./sec. \quad (7A\text{–c})$$

Remembering that energy is provided at a constant rate per unit area, Expressions 7A may be integrated between the limits o to l for x, and for S, between the initial thickness of film S_o and the final thickness S_{hl}. The initial film thickness S_o is determined by the liquid feed rate according to Equ. 2A. On performing the integrations we find that, for the whole length l of unit width, the energy expressions are

$$\Lambda f(5.83) \times 10^{-2} p. \sqrt{\frac{M}{T_s}}\, l \quad cal./sec. \qquad (8A\text{–a})$$

$$= \frac{\Lambda . \rho^2_{mh} g}{3\eta_{mh}}(S_o^3 - S_{hl}^3) \quad cal./sec. \qquad (8A\text{–b})$$

$$= k\frac{(t_h - t_s)}{S} l \quad cal./sec. \qquad (8A\text{–c})$$

The corresponding rate of evaporation in gm./sec. may be obtained if Expressions 8A–a and 8A–b are divided by the latent heat of vaporisation.

In expression (8A–b) the liquid density ρ_{mh} and the liquid viscosity η_{mh} have been assumed constant at a mean value, corresponding to a mean temperature. In practice, because both t_s and the temperature gradient $(t_h - t_s)/S$ are constants, t_h must vary with the film thickness S, from a value t_{ho} for S_o to a value t_{hl} for S_{hl}. Moreover, owing to the parabolic nature of the velocity distribution across the liquid film, the mean temperature across the film at any level is represented approximately by $(\frac{3}{4}t_h + \frac{1}{4}t_s)$ so that, for purposes of calculation, the values of ρ_{mh} and η_{mh} should be evaluated at the overall mean temperature

$$t_{mh} = \left\{\tfrac{3}{8}(t_{ho} + t_{hl}) + \tfrac{1}{4}t_s\right\}$$

For liquids in the lower vapour pressure range, it is necessary to maintain the liquid surface temperature t_s at a comparatively high value in order to achieve a reasonable rate of evaporation. In these circumstances the heat lost by radiation becomes appreciable and must be taken into consideration. The heated liquid surface, held at a temperature t_s, radiates approximately as a black body, so that the net rate of transfer of radiation between t_s and the condensed liquid surface at temperature t_c, for a length l of unit width, is given by

$$1.37 \times 10^{-12}(T_s^4 - T_c^4)\, l \quad cal./sec. \qquad (9A)$$

where T_s and T_c are in °K.

The heat flow according to Expression 9A, when added to the heat requirements represented by Expressions (8A–a) or (8A–b), gives the total heat flow represented by Expression (8A–c) and results in a compensating increase in the temperature t_h of the heating surface.

(*iv*) Heat Transfer in Condensing Falling Films

For the weight rate of condensation without re-evaporation on a length l of condenser surface of unit width, remembering that the thickness of the condensed film is initially zero, we have:

$$f\,(5.83) \times 10^{-2}\,.p\,\sqrt{\frac{M}{T_s}}\,l\,\frac{d_h}{d_c}\ gm./sec. \qquad (10A\text{–a})$$

$$= \frac{\rho^2_{mc}\,g\,S^3_{cl}}{3\rho_{mc}}\ gm./sec. \qquad (10A\text{–b})$$

where ρ_{mc} and η_{mc} are values corresponding to the overall mean temperature t_{mc} of the condensed liquid, and d_h and d_c are the diameters of the hot and cold surfaces.

The heat abstracted by the cooling water through the condenser wall, assumed to be held at constant temperature t_w, consists of the latent heat of condensation according to Expression 8A–a, and the heat lost by the condensate in cooling from the surface temperature t_s of the evaporating liquid to the mean temperature t_{mc} of the condensed liquid. If the mean specific heat of the condensate is c, then the rate of heat removal by the condenser wall per unit width is

$$\left\{\lambda + c\,(t_s - t_{mc})\right\}\frac{\rho^2_{mc}\,.g.\,S^3_{cl}}{3\,\eta_{mc}}\ cal./sec. \qquad (11A\text{–a})$$

$$= k\,\frac{(t_c - t_w)}{S}\,l\ cal./sec. \qquad (11A\text{–b})$$

The overall mean temperature t_{mc} of the film of condensed liquid may be approximated by assuming that

$$t_{mc} = \tfrac{1}{8}t_{cl} + \tfrac{7}{8}t_w$$

on the same basis as that adopted for calculating t_{mh}. The thermal radiation received by the condensed liquid is represented by Equ. 9A and results in a compensating increase in the temperature t_c of the liquid surface.

RATE OF EVAPORATION

Rate at which molecules of gas in equilibrium strike unit area is given by

$$\frac{\nu\bar{c}}{4}\ \text{molecules per second per cm.}^2 \qquad (12A)$$

where ν is the number of molecules per cm^3 and $\bar{c}$ is the mean velocity in cm./sec. of a typical molecule.

If the 'perfect gas' laws are assumed to apply, then Expression 12A may be written

$$5.83 \times 10^{-2} \times p_{mm}\sqrt{\frac{M}{T}}\ \ gm./sec.\text{–}cm.^2 \qquad (13A)$$

where p_{mm} is the equilibrium pressure, mm. Hg; M is the molecular weight, and T is the absolute temperature, °K.

Rate of evaporation from a liquid surface into a space, from which other molecules have been removed, is

$$5.83 \times 10^{-2} \times p_{mm}\sqrt{\frac{M}{T}}f\ \ gm./sec.\text{–}cm.^2 \qquad (14A)$$

where f is the evaporation coefficient.

MEAN FREE PATH

If it is assumed that the perfect gas laws apply, then the mean free path in equilibrium vapour is

$$\lambda_E = \frac{2.3 \times 10^{-20} \times T}{p_{mm} \times \delta^2}\ cm. \qquad (15A)$$

where T = temperature °K; p_{mm} = equilibrium vapour pressure, mm. Hg, δ = diameter of molecule, cm.

DIAMETER OF MOLECULE

If a close-packed structure is assumed for the liquid, then the diameter of a molecule is

$$\delta = 1.33 \times 10^{-8}\left(\frac{M}{\rho}\right)^{\frac{1}{3}}\ cm. \qquad (16A)$$

where M = molecular weight, and ρ = density of liquid, gm./cm^3.

SOME USEFUL EQUIVALENTS

Under equilibrium conditions there are, in the vapour phase

$$\left.\begin{aligned} & 1.60 \times 10^{-5} \times \frac{p_{mm}}{T}\ \ gm.\text{–}mol./cm.^3 \\ \text{or}\quad & 1.60 \times 10^{-5} \times \frac{M\,p_{mm}}{T}\ \ gm./cm.^3 \\ \text{or}\quad & 9.65 \times 10^{18} \times \frac{p_{mm}}{T}\ \ molecules/cm.^3 \end{aligned}\right\} \qquad (17A)$$

Flow rates of gases ($1\mu = 10^{-3}$mm.Hg):—

$$\left.\begin{aligned} gm./sec. &= litres\text{–}\mu/sec. \times 1.60 \times 10^{-5} \times \frac{M}{T} \\ &= c.c.\text{–}mm./sec. \times 1.60 \times 10^{-5} \times \frac{M}{T} \end{aligned}\right\} \qquad (18A)$$

Molecules in a monomolecular layer:—

$$6.54 \times 10^{15} \times \left(\frac{\rho}{M}\right)^{\frac{2}{3}} \text{ molecules/cm.}^2 \qquad (19A)$$

where ρ is the density of the condensed phase, gm./cm^3.

DIFFUSION

The following method of treatment was used by the author when discussing the process of gas evolution from low-vapour pressure liquids.*

If the diffusivity D is independent of concentration, as for gases in dilute solution in liquids, then the basic diffusion equation may be written

$$\frac{\partial C_l}{\partial \tau} = D\frac{\partial^2 C_l}{\partial x^2} \qquad (20A)$$

in which C_l is the gas concentration in the liquid at time τ at a distance x below the surface; at the liquid surface $x = 0$.

* See Trans. Inst. Chem. Engrs., **32,** No. 2, 1954, 99.

If the liquid extends an indefinite distance away from the surface and is initially free of gas, and if, at $\tau = 0$, gas at concentration C_g is brought to, and maintained in contact with, the liquid surface, then

$$C_l = C_s \left\{1 - erf \frac{x}{2\sqrt{D\tau}}\right\} \qquad (21A)$$

where C_s is the saturation—or equilibrium—value or gas concentration in the liquid at $x = o$, remembering that the solubility $L = C_s./C_g$.

Also, where the liquid is initially free of gas, the diffusion flow expressed as quantity of gas per unit time per unit area of liquid surface is

$$\mathcal{J} = -D\left(\frac{\partial C_l}{\partial x}\right)_{x=o} = C_s\sqrt{\frac{D}{\pi\tau}} \qquad (22A)$$

The total quantity of gas per unit area that has passed during time τ_s is given by

$$Q = \int_o^{\tau_s} \mathcal{J}\, d\tau = 2\, C_s \sqrt{\frac{D\,\tau_s}{\pi}} \qquad (23A)$$

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GASEOUS HEAT CONDUCTION AT LOW PRESSURES AND TEMPERATURES

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Summary

OVER THE LARGE TEMPERATURE DIFFERENCES that can exist in vacuum insulation of cryogenic devices some of the assumptions on which the formulae for free molecule heat conduction are based are not strictly valid. The consequences of this are discussed, and some incorrect usages are pointed out. Working formulae and auxiliary data on mean free paths and accommodation coefficients at cryogenic temperatures are presented.

Sommaire

LES SUGGESTIONS, sur lesquelles la formule de la conduction de la chaleur entre molécules libres est basée, ne sont pas strictement valides dans le domaine des grandes différences de température existant dans les dispositifs cryogèniques à isolément par le vide. L'auteur en discute les conséquences et quelques usages incorrects sont signalés. Enfin l'auteur présente des formules de travail et des données auxiliaires sur les libres parcours moyens et les coefficients d'accomodation pour les températures cryogéniques.

INTRODUCTION

THE USE OF HIGH VACUUM as an element of thermal insulation is familiar through the example of the Dewar vessel. In such devices it is not always practical to achieve such a high vacuum as to make the gaseous conduction of heat negligible compared to radiation. Indeed, when both boundaries are at a low temperature—as in a vessel for liquid hydrogen or helium when surrounded by liquid nitrogen or liquid air—the gaseous conduction may predominate. Consequently the calculation of such conduction is a necessary part of the engineering design of vacuum insulations.

The conduction of heat by gases is commonly separated into two distinct cases, namely, $L \ll d$ and $L \gg d$, where L is the mean free path in the gas and d is the distance apart of the boundaries between which the gas is transporting heat. The first of these is found at ordinary pressures and involves the thermal conductivity coefficient of the gas. The second case, free-molecule conduction, usually prevails in high-vacuum insulation, though occasionally the vacuum will be sufficiently poor so that an intermediate condition prevails.

The formulae for calculating free-molecule heat conduction are well known. However, the boundary temperatures are usually widely different in cryogenic insulations. This condition favours violation of some of the assumptions involved in these formulae, namely, that the distribution of molecular velocities following collision with a given boundary is Maxwellian and that the specific heat of the gas is constant.

Because of these departures the conduction formulae will be re-examined and practical procedures for applying them in engineering calculations will be pointed out. Several topics which are not now impor-

* MS. received January, 1958.

tant to the practising engineer are nevertheless introduced for the sake of completeness, but have been relegated to an Appendix.

THE HEAT CONDUCTION EQUATION

In textbooks, the derivation of the equation for free-molecule heat conduction is often carried out only for the simplified case of a surface exchanging energy with an equilibrium gas at a different temperature. Such a result is applicable, for example, to a heated wire in a bulb of such large diameter relative to the wire that the gas molecules come to effective equilibrium with the bulb through repeated collisions with it before each collision with the wire. However, in vacuum insulations, the two principal surfaces between which the gas is transporting energy are of comparable area and hence it is not possible for the gas to come to full equilibrium with either surface by repeated collisions. A satisfactory derivation of an equation for this more general case has been given by Kennard[1] and will be used as a basis for this discussion. This derivation is outlined in the Appendix. The result can be put in a form that is applicable to concentric spheres, coaxial cylinders, or parallel planes and is as follows:

$$H = a\left(\frac{\gamma+1}{\gamma-1}\right)\sqrt{\frac{R}{8\pi}}\frac{P}{\sqrt{MT}}(T_2 - T_1) \qquad (1)$$

$$a = \frac{a_1 a_2}{a_2 + a_1(1-a_2)A_1/A_2} \qquad (2)$$

where

- H is net energy transfer per unit time per unit area of inner surface
- γ is c_p/c_v, the specific heat ratio of the gas, assumed constant
- R is the molar gas constant
- P is the pressure
- M is the molecular weight of the gas
- T is absolute temperature
- a is the overall accommodation coefficient
- A is area and subscripts 1 and 2 refer to the inner and outer surfaces, respectively.

The constant, $\sqrt{R/8\pi}$, has the value 0.2426 for H in watt cm.$^{-2}$, P in mm. Hg, and T in degrees K.

Meaning of P and T

In the literature, T in the denominator of Equ. 1 is sometimes left undefined and is sometimes incorrectly taken to be the average temperature. In the authoritative treatise by Dushman[2] it is identified in one place as the temperature of the colder boundary and in another as the temperature of the outer boundary (for cylindrical configuration). These definitions are both incorrect in addition to being sometimes in conflict. The derivation of Equ. 1 makes it clear that T is an effective 'temperature' of the non-equilibrium gas which has a value intermediate between T_1 and T_2 but is not the average. (See Appendix, Item 2.)

The meaning of P also needs to be examined, since, under the conditions for free-molecule heat conduction, the thermal transpiration effect operates and the local pressure varies with local temperature throughout the evacuated space according to the law, $P/\sqrt{T}$ = constant[1]. In the derivation of Equ. 1, P is associated with T and, since $P/\sqrt{T}$ appears identically as a factor in Equ. 1, it is only necessary that values of P and T appropriate to the same point in the vacuum system are inserted into the formula. In principle P could be measured by a suitable gauge located between the walls where the effective gas temperature is indeed T. In practice, however, a value of P appropriate to an external gauge at ambient temperature is used. If the condition, $L \gg d$, also applies to the passage connecting the gauge to the vacuum space (where d is now the passage width), then *the temperature at the pressure gauge should be inserted for* T. This will be true even when this temperature lies outside the limits T_1 and T_2.

Variable γ

Over the large differences of temperature that are frequently involved in cryogenic insulations, γ for some gases may vary appreciably. For example, for hydrogen it varies from 1.63 to 1.40 between 80° and 300°K.

From the derivation of Equ. 1 it is clear that an average value of γ given by $\overline{c_p}/\overline{c_v}$ should be used where

$$\overline{c_p} \equiv \int_{T_1}^{T_2} c_p dT/(T_2 - T_1), \text{ etc.}$$

This is not identical with

$$\int_{T_1}^{T_2} \gamma \, dT / (T_2 - T_1),$$

though it is nearly the same for any practical case.

Mixture of Gases

Since by definition of the free-molecule mode of conduction the gas molecules interact predominantly with the walls rather than with each other, the conduction by a mixture is the sum of the conductions calculated for each component by Equ. 1 using for P the respective partial pressures.

TRANSITION REGION OF PRESSURE

Unfortunately there is no soundly-based formula for heat conduction that is valid at all pressures. The calculation may be extended a small way from the region where the ordinary thermal conductivity prevails toward lower pressures by introduction of the temperature discontinuity coefficient, but the entrance of the intermediate pressure region from the low pressure side where free-molecule conduction prevails has not been analysed. Two apparent exceptions have been noted in the literature:

(1) An equation due to Kistler[3] of a form which appears to bridge the transition region has been used to calculate heat transfer in vacuum-insulated vessels[4,5]. This equation can be written as follows:

$$K = 0.058 \sqrt{\frac{M}{T}} c_v L P \left(\frac{d}{d + \left[\frac{2 - a_o}{a_o} \right] L} \right) \quad (3)$$

where d and L are in cm., T is in °K, P is in mm.Hg, and K the apparent thermal conductivity is in watt/cm-deg K or cal/sec-cm-deg. K according to whether c_v is expressed in j/g-deg. K or cal/g-deg. K. The factor 0.058 is $1/\sqrt{2\pi R}$. The quantity a_0 is the accommodation coefficient, assumed to be the same for both walls, ($a_0 = a_1 = a_2$).

Kistler derived this formula for a random porous structure having mean pore size d and applied it to the flow of heat in silica aerogel. Use of the formula for calculating conduction across vacuum insulation has been justified on the basis that the formula should still hold for a uniform structure characterised by a boundary spacing equal to the aforementioned parameter d. Accepting this assumption, we can test the validity of the formula by seeing if it reduces to accepted forms at high and low pressures.

At high pressures ($L \ll d$) the bracketed factor is unity and the equation should correspond to the kinetic theory expression for the ordinary thermal conductivity,

$$K = \epsilon \eta c_v \quad (4)$$

in which ϵ is a numerical factor, and η is the viscosity.

Equ. 3 can be put in the form of Equ. 4 by using well-known relations as follows,

$$0.058 = 1/\sqrt{2\pi R}$$
$$L = \eta / 0.499 \, \rho \, \bar{v}$$

$$\rho = MP/RT \quad (5)$$
$$\bar{v} = \sqrt{8 \, RT/\pi M} \quad (6)$$

where ρ is the density and $\bar{v}$ is the mean velocity.

The result is

$$K = \eta c_v / (4 \times 0.499)$$

It is smaller than Equ. 4 by the factor 0.499 (4 ϵ) which for ordinary gases ($\epsilon \simeq 2$) amounts to about four.

Similarly we can see if Kistler's equation reduces to Equ. 1 at low pressures ($L \gg d$). Parallel configuration is assumed and the following relations are used:

$$0.058 = 1/\sqrt{2\pi R}$$
$$H = K (T_2 - T_1) / d \quad (7)$$

$$c_v = R/M (\gamma - 1) \quad (8)$$
$$a = a_o / (2 - a_o)$$

The result is then seen to have the same form as Equ. 1 except that it is smaller by a factor $(\gamma + 1) / 2$. This discrepancy is readily traceable to the fact that Kistler in effect takes $c_v T$ as a measure of the heat content per gram of gas molecules effusing from an equilibrium body of gas at temperature T whereas the correct expression can be shown* to be $(c_v + R/2M)T$.

*See, for example[1], pp. 312–3.

Thus Equ. 3 does not reduce to accepted formulae in the limits of high or low pressures. Inspection shows no reason to suppose that it is accurate at intermediate pressures in the present application.

(2) Dushman[6] has given equations for the transition region utilising the temperature discontinuity coefficient symbolised by g. This discontinuity appears at the boundary of a body of gas across which a temperature gradient is impressed as the pressure is lowered from the region of ordinary conduction into the transition region. The coefficient, g, is a length

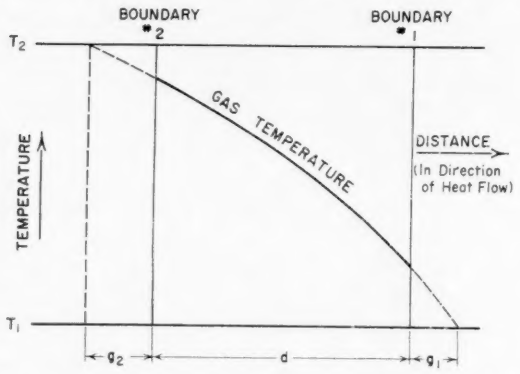

Fig. 1. Representation of the temperature discontinuity coefficients.

of the order of magnitude of L. The formulae for ordinary conduction are modified for this effect by imagining each boundary to be moved back by the distance, g (Fig. 1). Thus for parallel configuration

$$H = \frac{\overline{K}(T_2 - T_1)}{d + g_1 + g_2} \tag{9}$$

and for cylindrical configuration

$$H = \frac{\overline{K}(T_2 - T_1)}{r_1 \ln \dfrac{r_2 + g_2}{r_1 - g_1}}$$

$$= \frac{\overline{K}(T_2 - T_1)}{r_1 \left[\ln \dfrac{r_2}{r_1} - \ln\left(1 - \dfrac{g_1}{r_1}\right) + \ln\left(1 + \dfrac{g_2}{r_2}\right) \right]}$$

Provided that $g_1 \ll r_1$, $g_2 \ll r_2$, (and the above equation is meaningless without this restriction) then for cylinders

$$H \simeq \frac{\overline{K}(T_2 - T_1)}{r_1 \left(\ln \dfrac{r_2}{r_1} + \dfrac{g_1}{r_1} + \dfrac{g_2}{r_2} \right)} \tag{10}$$

Dushman shows that, if $g_1 = g_2$, Equ. 9 reduces to the Knudsen formula Equ. 1 in the low pressure limit. However, the assumption of equality of conditions at the two boundaries limits his result to cases where $T_2 - T_1$ is small. Equ. 10 would not be expected to apply in the low pressure limit and in fact does not reduce to Equ. 1. Dushman's success in fitting the formula to data by Knudsen covering a wide range of pressures* should therefore be regarded as a fortunate coincidence due in part to the fact that the data cover only a narrow temperature range. The expression yields an incorrect value of α_1.

In his derivation of an expression for g, Kennard[7] makes it clear that the concept of the temperature discontinuity coefficient is applicable only near the high pressure end of the transition region. Nevertheless expressions having the form of Equs. 9 or 10 but regarded as empirical, may be useful for approximate calculations in the transition region. For example we may insert in Equ. 9 Kennard's expression for g, which with the aid of Equs. 4, 5, 6 and 8 we will put in the following form:

$$g_i = \frac{K_i}{P} \frac{2 - \alpha_i}{\alpha_i} \frac{\gamma_i - 1}{\gamma_i + 1} \sqrt{\frac{2\pi M T_i^*}{R}} \quad (i = 1, 2)$$

Now if K_1, γ_1 and T_1^* are taken to be suitably defined averages of these properties in the region of temperature discontinuity next to boundary 1 and similarly for the quantities in the analogous expression for g_2, then Equ. 9 becomes identical with Equ. 1 in the low pressure limit where the 'slip' regions merge. The expression thus is of reasonable form though its accuracy in the transition region will be questionable. If T_2 and T_1 are greatly different, the values of K_i and γ_i will depend on T_i^* and all three must be found by trial.

The representation of the transition region by

*See[2] pp. 57–8.

Equ. 9 is equivalent to multiplying $\overline{K}$ by the factor $d/(d + g_1 + g_2)$ which for diatomic gases, small temperature difference, and unit accommodation coefficient is approximately $d/(d + 4L)$. By contrast, Kistler's equation applied to the same conditions is equivalent to multiplying a value of $\overline{K}$ that is four times too small by a transition factor $d/(d + L)$.

Mean Free Paths

To facilitate determination of the mode of conduction Table I shows selected values of mean free path.

Table I. **Selected Values of Mean Free Path**
(L in cm. at P = 1 micron Hg)

Temperature	4°K	20°K	77°K	300°K
Air	—	—	0.87	5.1
Hydrogen ..	—	0.30	1.8	9.5
Helium ..	0.11	0.67	3.2	15.

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The mean free path is conveniently obtained from tabulated values of viscosity[8] using the formula,

$$L = 8.6 \frac{\eta}{P} \sqrt{\frac{\overline{T}}{M}}$$

where L is in cm., T is in deg K, η is in poises and P is in mm.Hg.

Experimental Values of Accommodation Coefficients

The definition of accommodation coefficient will be considered more fully in the Appendix. For engineering purposes it is sufficient to consider it as an empirical factor needed to bring conduction as computed by Equs. 1 and 2 into agreement with experimental values. It is determined using configurations such that $A_1/A_2 \ll 1$ and hence $a \simeq a_1$, which permits isolation of the value for the inner of the two boundary surfaces. Accommodation coefficients for light gases are of special interest as they can have rather low values. Experimental studies have shown two types of behaviour.

For metallic surfaces that have been flashed in vacuum to remove adsorbed films, the initial accommodation coefficient can be strikingly small—only a few per cent. for helium on tungsten[9]—and it decreases with decrease in temperature, provided the temperature is not so low that an adsorbed film of the gas or a condensed layer forms immediately. The interaction between a gas molecule and a clean solid surface has been treated quantum-mechanically[10].

On the other hand, surfaces that have received no special treatment to remove films show larger values that increase toward unity as the temperature is lowered.

It will not escape notice that the first type of behaviour is ideally suited to cryogenic insulation since the insulation would then be better and would improve (up to a point) in the temperature direction in which the cost of refrigeration increases. However, this behaviour has not yet been realised with other than filaments, and the second type of behaviour must be regarded as typical of the unflashed surfaces of large Dewars. The suggested values in the Table II were derived from the meagre low temperature data of the second type[11–15].

It is interesting to note that the low values for the light gases at temperatures above 80°K counteract the effect of the factor $1/\sqrt{M}$ in Equ. 1 and thus result in conduction of the same magnitude as by air at the same pressures. This is in marked contrast to the case at atmospheric pressure where the conductivities K of hydrogen and helium are about an order of magnitude greater than that of air.

As Knudsen showed, a_1 is increased by surface roughness, due to the increased probability of multiple collisions. Rowley and Bonhoeffer[15] found the interesting result in the region around 150°K that

Table II. **Suggested Values of Accommodation Coefficient** a_1

Temperature (°K)	*Helium*	*Hydrogen*	*Air*
300	0.3	0.3	0.8–0.9
80	0.4	0.5	1
20	0.6	1	
4	(1)		

a_1 for para-hydrogen is 10 to 15% smaller than for normal hydrogen. This was attributed to the accommodation coefficient for rotational energy (which is a larger part of the total energy in the *para* form) being less than the accommodation coefficient for translational energy.

The experimental values have nearly all been obtained at gas pressures of 10 microns or more, since, in working with filaments, free-molecule conduction predominates when the mean free path exceeds the filament diameter, and the latter is quite small. On the other hand, the pressures required in vacuum insulation on an ordinary scale are usually 0.1 micron or less. The pressure variation of a_1 for various gases on platinum near ambient temperature has been measured by Amdur, Jones, and Pearlman[16] for pressures in the range 10 to 500 microns. The values are comparable to those in the above table but show small increases with increasing pressure. Amdur[17] has shown that this variation can be accounted for on the assumption that the accommodation coefficient is a linear function of the fraction of surface covered by adsorbed gas, the latter being given by Langmuir's adsorption isotherm. If his expression is used to extrapolate the accommodation coefficient to pressures of 0.1 micron or less, values of only a few per cent. are obtained, approaching the values mentioned previously for flashed surfaces. However, unpublished work at this laboratory at pressures down to 0.1 micron does not show such a decrease but rather indicates values conforming approximately to those in the table.

WORKING FORMULAE

We can put Equ. 1 in the following form

$$H = (\text{constant})\ a\ P\ (T_2 - T_1)$$

where the constant is

$$\frac{\gamma + 1}{\gamma - 1}\sqrt{\frac{R}{8\pi MT}}$$

and is listed below for various gases using $T = 300°\text{K}$ as the assumed temperature at the pressure gauge, H in watts cm.$^{-2}$ of inner surface, P in mm.Hg, and temperature in deg. K.

Table III

Gas	T_2 *and* T_1 *in* °K	*constant*
N_2	$\leqslant 400$	0.0159
O_2	$\leqslant 300$	0.0149
H_2	300 and 77 or 300 and 90	0.0528
H_2	77 and 20	0.0398
He	any	0.0280

APPLICATION TO DEWARS

Heat leaks in Dewar vessels fall into three classes: Radiation, conduction in solid supporting members and piping, and gas conduction. Conduction in supports and piping can be made negligible provided the vessel is not required to be exceptionally compact or to withstand rough handling. Consequently the significance of the gas conduction can be seen by comparing its magnitude with that of the radiation heat leak.

Consider two practical examples: (*a*) A Dewar for liquid helium in which a thermal shield maintained at the temperature of boiling nitrogen (77°K) is interposed between the helium container and the outer shell of the vacuum space, and (*b*) a similar vessel for liquid hydrogen. Radiation decreases much more rapidly with decreasing temperature than does gas conduction. Thus the gas conduction will be a more significant part of the total heat leak from the shield to the inner vessel than of the heat leak from ambient temperature to the shield.

In comparing the radiation with the gas conduction from shield to inner vessel we will assume emissivities of 0.03 for all surfaces and parallel plate configuration. The heat leak due to radiation is then found to be 3×10^{-6} watt/cm.^{2} for either of the above cases. In calculating the gaseous heat conduction we will use accommodation coefficients from the preceding table but must first decide what kind of gas is present.

(*a*) A surface at the temperature of boiling helium (4°K) is an efficient condenser for all volatiles other than helium. Thus helium is the only gas that can maintain itself in the insulating vacuum of a helium Dewar. (*b*) A surface at the temperature of boiling hydrogen (20°K) is an efficient condenser for all common atmospheric gases except helium, hydrogen, and neon. Of these, hydrogen should be predominant due either to leakage from the liquid container or evolution from metals in the system. Thus hydrogen will be assumed to account for the gas conduction in a Dewar for liquid hydrogen.

If we calculate from Equ. 1 or the derived working formula the gas pressure (measured at ambient temperature), at which the gas conduction from the 77°K shield to the inner vessel equals the radiation, we obtain (*a*) 4×10^{-6}mm.Hg for a helium Dewar, and (*b*) 3×10^{-6}mm.Hg for a hydrogen Dewar. (These pressures are well within the region where Equ. 1 is applicable to vessels of ordinary size, since the various g values are found to exceed 100 cm. even at 10^{-5}mm.Hg.) Thus it will be seen that such vessels require a rather high order of vacuum technique on the part of the manufacturer.

An interesting footnote is provided by the observation that commercial metal Dewars of the above type often have several-fold smaller heat leak when containing liquid helium than when containing liquid hydrogen[17a]. If the heat leak when containing helium is attributed to radiation, calculation shows that the surfaces must have excellent emissivity values, i.e. 0.02 or less. Consequently there can be but negligible gas conduction in this condition and the vessels must be free from leaks into the vacuum space. The higher heat leak when containing liquid hydrogen must then be attributed to hydrogen evolved into the vacuum space from the metals of construction following sealing off of the vacuum space by the manufacturer.

* Considering the particles as deBroglie waves, it can be shown that technical surfaces are far too rough to give appreciable specular reflection and this has been amply verified experimentally. See Ref.[10] pp. 594–6 or L. B. Loeb, *Kinetic Theory of Gases*, McGraw-Hill Book Co., 1934, pp. 331–4.

APPENDIX

(1) Derivation of Heat Flow Equation

For reference in the discussion to follow, the derivation of Equ. 1 will be given briefly. Let us define E_1' as the energy content of the molecules that leave unit area of Surface 1 each second, and E_2' as the energy content of the molecules that leave unit area of Surface 2 each second (Fig. 2). If the emission of gas molecules from Surface 2 is diffuse, *i.e.* obeying a cosine law like Lambert's law in optics, it can be shown for cylindrical or spherical symmetry

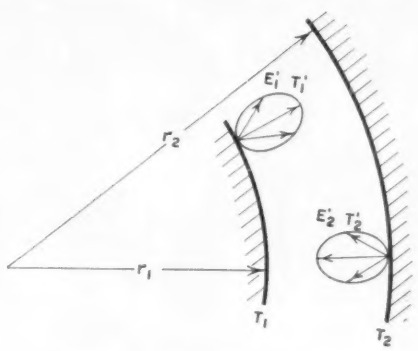

Fig. 2. Schematic drawing identifying the two effusing gas streams that perform free-molecule heat conduction.

that the fraction of stream 2 striking Surface 1 is A_1/A_2*. Then the energy brought up to unit area of Surface 1 per second is

$$E_2'\ A_2\ (A_1/A_2)\ /\ A_1 \text{ or simply } E_2'.$$

The energy brought up to unit area of Surface 2 per second is

$$E_1'\ A_1/A_2 + E_2'\ (1 - A_1/A_2).$$

The accommodation coefficients are expressed as fractions of the maximum possible energy exchange at the respective walls:

$$E_2' - E_1' \equiv \alpha_1\ (E_2' - E_1) \qquad (11)$$

$$E_1'\ A_1/A_2 + E_2'\ (1 - A_1/A_2) - E_2' \equiv \qquad (12)$$

$$\alpha_2\ [E_1'\ A_1/A_2 + E_2'\ (1 - A_1/A_2) - E_2]$$

where E_1 and E_2 are the values that E_1' and E_2' would have if thermal equilibrium were achieved in all collisions at the surfaces. The net energy transfer between the surfaces is

$$H \equiv E_2' - E_1' = \alpha(E_2 - E_1)$$

and a is given by Equ. 2. E_2 and E_1 are the same as the energy contents of molecules effusing from equilibrium bodies of gas at T_2 and T_1 respectively, and crossing unit area of boundary. The mean translational energy of such molecules[1] is $2kT$, whence the mean total energy per gram is

$$\int_o^T (c_v + R/2M)\, dT.$$

If S_1 and S_2 are defined as the mass rates of emission of molecules from unit areas of Surfaces 1 and 2 respectively, then

$$E_1 = S_1 \int_o^{T_1} (c_v + R/2M)\, dT \quad (13), \text{ etc.}$$

But from the mass flow balance at Surface 1

$$S_1 A_1 = S_2 A_2 \, (A_1/A_2)$$

or $$S_1 = S_2 \equiv S_o$$

Thus $$E_2 - E_1 = S_o \int_{T_1}^{T_2} (c_v + R/2M)\, dT$$

At this point c_v is assumed to be constant, whence

$$E_2 - E_1 = S_o(c_v + R/2M)\,(T_2 - T_1)$$

$$= \frac{S_o R\,(\gamma + 1)}{2M\,(\gamma - 1)}(T_2 - T_1)$$

Now the emission (or impact) rate per unit area is not merely the same at the two boundaries but also for any element of surface located arbitrarily in the gas. This can be shown as follows: S at the chosen element of surface is the sum of impacts by molecules arriving from the Boundaries 1 and 2. Therefore it is S_1 times a configurational factor given by a definite integral, plus S_2 times another configurational integral or

$$S = a\, S_1 + b\, S_2 = (a + b)\, S_o$$

where a and b are the respective configurational factors.

Now if $T_1 = T_2$ we will have that all S's are equal because of the requirement of pressure equilibrium. Thus

$$a + b = 1$$

But a and b depend only on configuration and therefore $(a + b)$ must continue to be unity when $T_1 \neq T_2$. Thus $S = S_o$ in general.

It will be convenient then to express S_o as the impact rate at the boundary of a hypothetical equilibrium gas having the same mean density and mean molecular velocity as our actual gas. Thus from kinetic theory[1],

$$S_o = \rho\bar{v}/4 \quad (14)$$

Making the further conventional substitutions, (5) and (6), we arrive at Equ. 1.

(2) The Effective Gas Temperature

It was pointed out earlier that evaluation of the effective gas temperature T is not usually necessary due to the circumstance that the value assumed for P is usually not the true internal pressure but rather is the pressure in a lobe of the vacuum system that is at ambient temperature. Nevertheless, for completeness we may examine the definition of T. First S must be expressed in the form of Equ. 14 but in terms of the mean partial densities, ρ_1 and ρ_2, and mean velocities $\bar{v}_1$ and $\bar{v}_2$ of the corresponding streams of gas. The factor $1/4$ would apply for the impact rate from a body of gas with random molecular motion, *e.g.* in expressing the impact rate on Surface 1 when $A_1/A_2 \ll 1$. On the other hand, for parallel configuration $(A_1/A_2 = 1)$, the molecules in stream 2 have velocity components in the direction of Surface 1 but none are directed away from it, while those in stream 1 are directed toward Surface 2 but none away from it. In this case, each stream is one half of a random population, the impact rate is twice as high because of the favourable selection of velocity components, and the factor in Equ. 14 is $2 \times 1/4 = 1/2$. For intermediate configurations a geometric factor must be introduced which will provide the necessary transition. Thus we assume for the impact rate on Surface 1

$$S = \rho_2\bar{v}_2\,(A_1/A_2 + 1)\,/4 \quad (15)$$

This must equal the emission rate from Surface 1 which can be written as one-half $\bar{v}_1$ times the density of stream 1 as it leaves Surface 1. Since this stream is divergent its density at Surface 1 is greater than the mean density ρ_1 by the factor $(A_1 + A_2)/2A_1$. Hence the emission rate is

$$S = \rho_1\bar{v}_1\,(A_2/A_1 + 1)\,/4 \quad (16)$$

Also we have

$$\rho = \rho_1 + \rho_2 \quad (17)$$

Combining Equs. 14, 15, 16 and 17 we can eliminate ρ_1 and ρ_2 and express $\bar{v}$ as a function of $\bar{v}_1$ and $\bar{v}_2$. In addition we will use equations like 6 to replace the mean velocities by corresponding effective temperatures. The result is

$$\frac{1}{\sqrt{\bar{T}}} = \left(\frac{A_1}{A_1 + A_2}\right)\frac{1}{\sqrt{\bar{T}_1'}} + \left(\frac{A_2}{A_1 + A_2}\right)\frac{1}{\sqrt{\bar{T}_2'}}.$$

The quantities T_1' and T_2', which are effective temperatures of the gas streams leaving Boundaries 1 and 2, respectively, can be expressed in terms of the boundary temperatures T_1 and T_2. Since we have already assumed c_v to be constant, each E is proportional to the corresponding T and by replacing E's by T's throughout Equ. 11 and 12 we obtain

$$T_1' = \frac{a_1 T_1[(1-a_2)\,A_1/A_2 + a_2] + a_2(1-a_1)\,T_2}{a_2 + a_1(1-a_2)\,A_1/A_2} \quad (18)$$

$$T_2' = \frac{a_1 T_1(1-a_2)\,A_1/A_2 + a_2 T_2}{a_2 + a_1(1-a_2)\,A_1/A} \quad (19)$$

We may note that as $A_1/A_2 \to 0$ then $T \to T_2$, as would be expected since the gas must then approach thermal equilibrium with the enclosing Surface 2. This reasonable result does not follow from Kennard's treatment due to his neglecting the configuration factors in Equ. 15 and 16.

(3) Definition of Accommodation Coefficient

In the original development by Knudsen, the accommodation coefficients were defined analogously to Equ. 11 and 12 except that each E was represented by the corresponding T. Blodgett and Langmuir[18] pointed out that this was objectionable since in general the gas streams represented by E_1' and E_2' would not have a velocity distribution characteristic of gases in thermal equilibrium and hence could not be assigned temperatures in the usual thermodynamic sense. They introduced the usage of defining the accommodation coefficient directly in terms of energies equivalent to Equ. 11 and 12. However, Knudsen[19] referring to his definition has stated 'the temperatures . . . stand in fact instead of the mean energies', and indeed the two usages lead to the same Equ. 1 for heat conduction. The two developments can be compared in the following outline:
after Knudsen:

$$E_2' - E_1' = S(c_v + R/2M)(T_2' - T_1') \quad (20)$$

$$a \equiv (T_2' - T_1')/(T_2 - T_1)$$

$$\therefore E_2' - E_1' = S(c_v + R/2M)\,a\,(T_2 - T_1) \quad (21)$$

after Blodgett and Langmuir:

$$E_2' - E_1' \equiv a\,(E_2 - E_1)$$

$$E_2 - E_1 = S(c_v + R/2M)(T_2 - T_1) \quad (22)$$

$$\therefore E_2' - E_1' = S(c_v + R/2M)\,a\,(T_2 - T_1) \quad (23)$$

In the former, the coefficient $(c_v + R/2M)$ in the mean energy of unit mass of an effusive stream is based upon the assumption of a Maxwellian velocity distribution, yet it is identified with two streams at T_2' and T_1' known to be generally non-Maxwellian. On the other hand, in the second treatment, this factor is identified not with the actual gas streams (denoted by primes) but rather with hypothetical streams in full equilibrium with the respective walls at T_1 and T_2. These streams are Maxwellian and there is no objection to assigning them temperatures T_1 and T_2 in the usual sense. Thus it may be granted that the second procedure is logically the more satisfactory. However, the practical effect of the distinction in engineering calculations using experimental values of accommodation coefficient is nil since the same equation is used for heat transfer as for calculating the accommodation coefficients.

Oliver[20] has seized upon the idea of Blodgett and Langmuir and has implied that it avoids entirely any assumption of the Maxwellian distribution, and therefore that the resulting heat conduction equation is rigorously true for any values of $T_2 - T_1$, while the same heat conduction equation when derived with the usage of Knudsen is restricted to the limit $(T_2 - T_1) = 0$. That the former is not generally correct may be seen by examining the origin of Equ. 6. It will be found that the assumption of Maxwellian velocity distribution is involved here and that the expression in effect contains the factor $\sqrt{8/3\pi}$ which relates the average and rms. velocities in a Maxwellian gas*. The heat conduction equation thus will hold rigorously only if the gas is actually Maxwellian. This condition is met only for the special cases, $a = 1$, or $T_2 = T_1$, or $A_1/A_2 = 0$.

However, a real (though trivial) difference in the two approaches appears, if c_v is a function of temperature. It is found then that in Equs. 20 and 21 we must use the average value of c_v in the interval T_2' to T_1' while in Equs. 22 and 23 the average in the interval T_2 to T_1 appears. Other Equations such as 18 and 19, in which proportionality between E and T is involved, also will be modified through replacement of the proportionalities by relationships like 13.

(4) Accommodation Coefficient Dependent on Gas Temperature

It has often been pointed out that Equ. 1 or equivalent forms are not rigorously correct for the case of large $\triangle T$, and we have seen that this is true provided

* It might be thought that Equ. 14 also involves the Maxwell distribution since the latter is often introduced into its derivation. However, this is not essential, and Knudsen shows[21] that it is only necessary to assume that the molecular motions are randomly directed in space. We have already adopted the latter assumption in another connection.

$a \neq 1$ and $0 < A_1/A_2 < 1$ regardless of whether the accommodation coefficients are defined in terms of temperature or energy. Some experimental studies in which a_1 was measured while T_2 was varied widely[12,15] have shown no significant dependence and have been cited as indication that the failure of Equ. 1 that would be expected at large $\triangle T$ due to non-Maxwellian distribution has not materialised. However, it is incorrect to expect such failure for the condition, $A_1/A_2 \ll 1$, under which these experiments were performed, because stream 2 is in equilibrium with Boundary 2 and therefore is Maxwellian, while the distribution in stream 1 is unimportant since this stream is a negligible part of the total molecular population. What these experiments did show was that a_1 was apparently independent of the energy of the incident molecules. This suggests that it should be unnecessary to distinguish 'spectral' and 'total' accommodation coefficients*.

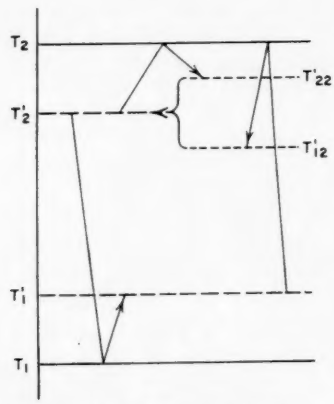

Fig. 3. Representation of the composite stream leaving Surface 2.

It would appear then that experiments in which the area ratio is near unity and $\triangle T$ could be made large would be useful in testing Equ. 1. These are conditions of practical importance since they correspond to the situation in large Dewars. They are also conditions under which Equ. 1 would not be expected to apply rigorously. Since such an experiment does not permit evaluation of the individual accommodation coefficients but only the overall factor, a, it would be necessary to determine the former in separate experiments in which the condition, $A_1/A_2 \ll 1$, would permit isolation of the accommodation coefficients for individual surfaces.

In case the latter experiments should disclose a dependence of accommodation coefficient on the mean energy (temperature) of the incident gas molecules, the calculation of a_2 by Equ. 12 would become more complicated, since the molecules incident on Surface 2 compose two groups which can have widely different mean energies in as much as they had their last previous collisions at Surfaces 1 and 2 respectively. This contingency requires consideration only under certain conditions. Thus, it is not of practical importance when a_1 and a_2 are both small, because then the mean energies corresponding to E_1' and E_2' are not widely different even though T_2 and T_1 are far apart. Also the case where a_1 and a_2 are both near unity need not be considered since a strong dependence of accommodation coefficient on incident energy is ruled out by definition. A case that would require consideration is when a_1 is near unity and a_2 is small, *e.g.* hydrogen with $T_2 = 77°\text{K}$, $T_1 = 20°\text{K}$, or helium with $T_2 = 77°\text{K}$, $T_1 = 4°\text{K}$. These cases arise with Dewars for liquid hydrogen or helium that are protected by a radiation shield at the temperature of liquid nitrogen. This situation is shown schematically by Fig. 3. The stream at temperature T_2' is a composite of molecules that have rebounded from Surface 2 after a previous collision with Surface 1 (labelled T_{12}') and molecules that have had both previous collisions at Surface 2 (labelled T_{22}'). We may define two additional accommodation coefficients for these processes:

$$a_{12} \equiv (E_1' - E_{12}')/(E_1' - E_{12})$$

$$a_{22} \equiv [E_2' \ (1 - A_1/A_2) - E_{22}']/[E_2' \ (1 - A_1/A_2) - E_{22}]$$

where E_{12}', E_{12}, E'_{22}, E_{22}, are defined analogously to E_1', E_1, E_2', E_2. Also

$$E_2' = E_{12}' + E_{22}' \tag{24}$$

We can replace the various E's by corresponding T's through substitutions of the form of Equ. 13 using the additional relations

$$S_{12} = S_1 \, A_1/A_2$$

$$S_{22} = S_2 \, (1 - A_1/A_2)$$

Of course, E_{12} and E_{22} correspond to T_2.

* A possible exception appears in a paper by Mann[22] who found a small increase for helium on previously flashed platinum at high temperatures as the gas temperature was varied from ambient to 80°K. The difference apparently vanished as the platinum temperature was reduced toward ambient. Oliver [20] found the opposite trend for several gases on tungsten.

If c_v is constant, Equ. 24 becomes

$$T_2' = T_{12}' A_1/A_2 + T_{22}' (1 - A_1/A_2)$$

From these relations, those defining a_1 and a_2, and experimental values of $a_1 = f(T_2')$, $a_2 = f(T_1')$ and c_v one can arrive at the values of T_1', T_{12}', T_{22}' T_2', and hence the composite accommodation coefficient a_2, by trial.

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an all-metal apparatus for THE DETERMINATION OF SPECIFIC SURFACE BY GAS ADSORPTION

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Summary

A GAS ADSORPTION APPARATUS is described in which the conventional glass items such as stopcocks and mercury-filled manometers are replaced by metal valves and multi-pointer capsule gauges. The apparatus can be used by semi-skilled operators to obtain complete adsorption isotherms, single or two-point comparison values, or rates of diffusion into micro-porous solids.

Sommaire

DESCRIPTION D'APPAREILS à adsorption de gaz dans lesquels les parties généralement en verre, les robinets d'arrêt et les manomètres à mercure sont remplacés par des vannes métalliques et des jauges à capsule à aiguilles multiples. Les appareils peuvent être employés, par des opérateurs non-spécialisés, pour obtenir une adsorption complète isotherme, ou des valeurs de comparaison à un ou deux points ou des taux de diffusion dans des solides microporous.

INTRODUCTION

ALL THE TYPES of glass apparatus used for the determination of specific surface by gas adsorption, described in Vol. III, No. 3 of this journal, have certain characteristics which render them unsuitable for continuous use by semi-skilled personnel. Some of these undesirable characteristics, such as slowness of operation, and the amount of skill required in manipulating the stopcocks and taking the readings, can be reduced by careful design and by adopting a simplified form of the apparatus, but the inherent fragility of the glass apparatus means that a moment's carelessness can still lead to severe damage which may take a considerable time to repair.

The apparatus described in this paper is essentially a double manometer, constant volume apparatus (see the schematic diagram, Fig. 1) in which the gauge and hollow copper block B comprise the volumetric dosing system, the gauge C and sample container A comprise the gas adsorption system, and D is the dosing valve between the two systems.

The detachable sample bulb is the only essential glass component; all the remaining items, with the exception of the drying trap, are of metal joined by O-ring unions and copper tubing.

Figs. 2 and 2a are views of the apparatus from the front and back respectively and Fig. 3 is a diagrammatic presentation of Fig. 2, in which all the major components and the valves occupy the same relative positions as in the photograph.

The cabinet shown is made from welded angle girders 3 ft. high $2\frac{1}{2}$ ft. wide and $1\frac{1}{2}$ ft. deep, but if preferred it could be constructed by bolting

* M.S. received June, 1957.

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together sections of slotted angle girder.

DESIGN OF THE APPARATUS

The construction is best explained by considering each section in turn:

Gas Storage and Handling

The gas used for surface area determinations is dichlorodifluoromethane (CF_2Cl_2 or Freon-12) which is stored, as supplied, in a 9-lb. cylinder behind the instrument panel at the top of the apparatus with the cylinder-head control valve (not visible in Fig. 2) projecting at the side of the framework to the left of the instrument panel. On opening the cylinder valve, liquid Freon flows into the bottom of an inclined reservoir filling the upper half of the reservoir with gas. Since this gas is at a pressure of about 70 p.s.i., and the gauges have a maximum rating of only 25 p.s.i., there is a danger that careless manipulation of the inlet valve to the gauge systems might result in damage to the gauges. To reduce this risk, a reducing valve set for 22 p.s.i. is inserted between the reservoir and the inlet valve.

If it is desired to use any other gas, such as nitrogen, hydrogen, oxygen or methane, the Freon cylinder can be detached at the O-ring Union, B (Fig. 3), and gas admitted to the reservoir, through a flexible armoured pipe after exhausting the reservoir and pipe with the rotary and oil diffusion pumps.

Dry air, nitrogen or helium for the determination of the dead space can be admitted separately through the air-inlet valve.

Volumetric Dosing System

Gas from the storage system, or the air-inlet valve, see Fig. 3, is admitted to the pressure gauge M_2 and the drilled copper block V_1 and V_2 through the isolating valve, while the baffle valve on the head of the diffusion pump is closed. All the valves in this section are $\frac{1}{4}$-inch bore diaphragm valves. The total volume of the system consists of the volume V_1 or $(V_1 + V_2)$ of the block, plus the volume of the tubing between the dosing valve, the block, the isolating valve and the gauge, together with the volume of the capsule in the gauge. Therefore, once these volumes are known the volume of gas at S.T.P. in the system can be calculated from the pressure reading and the temperature of the copper block. No temperature control of the system is employed; in practice it is found that after a one-hour run the whole of the metal work is at the same reasonably constant temperature. This temperature is indicated by a thermometer inserted in a hole drilled in the copper block, the stem being visible at the front of the instrument panel.

The copper block is made from a solid cylinder, about 15 cm. long and 5 cm. in diameter, in which two wells are drilled, of 25 and of 35 c.c. capacity.

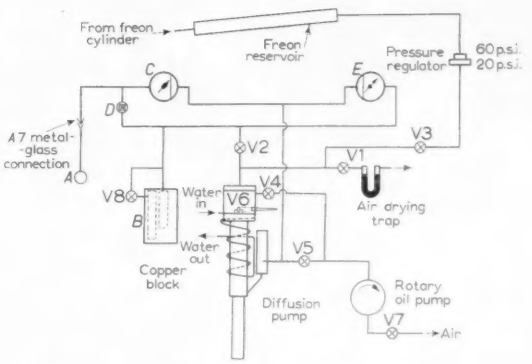

Fig. 1. Schematic layout of apparatus.

These holes are sealed off with copper discs brazed to $\frac{1}{4}$-inch i.d. copper tubing carrying $\frac{1}{4}$-inch O-ring unions for connection to the rest of the system. The large well is permanently open to the system, and the small well functions as an extra volume to be added to the system, when required, through an ordinary diaphragm valve.

The Adsorption System

Doses of gas are admitted to the bulb of the sample container, and gauge M_1, through the dosing valve which is a combined fine control and wide bore by-pass valve, the by-pass being disclosed when the fine control is fully opened. All connections in this system are kept as short as possible and are made from

1/16-inch bore 'hypodermic' tubing.

The sample container is connected to the copper tubing by an A7 cone and socket, the cone being turned and ground by hand from a short section of drilled brass rod. A satisfactory sample container is made by blowing a bulb of between 5 and 10 c.c. capacity on the end of a 14-cm. length of 4-mm. i.d. glass tubing and A7 socket, into which is slipped a 13-cm. length of 3-mm. diameter glass rod. This rod serves both to minimise the dead space and to retard 'fluidisation' of the sample. It is advisable to make a hook at the end of the glass rod, located in the bulge just below the socket enabling the rod to be withdrawn by means of a loop of copper wire. The A7 joint should be sealed with black wax if the sample is to be degassed above 250°C, otherwise a satisfactory seal can be made with a hard vacuum grease.

A small tubular furnace designed to operate between 50°C and 400°C, and controlled by a thermal switch is used for degassing the sample. For ease of operation it is counterpoised and slides on guide rails. When not in use as a heater it serves as a platform for the refrigerant Dewar vessel.

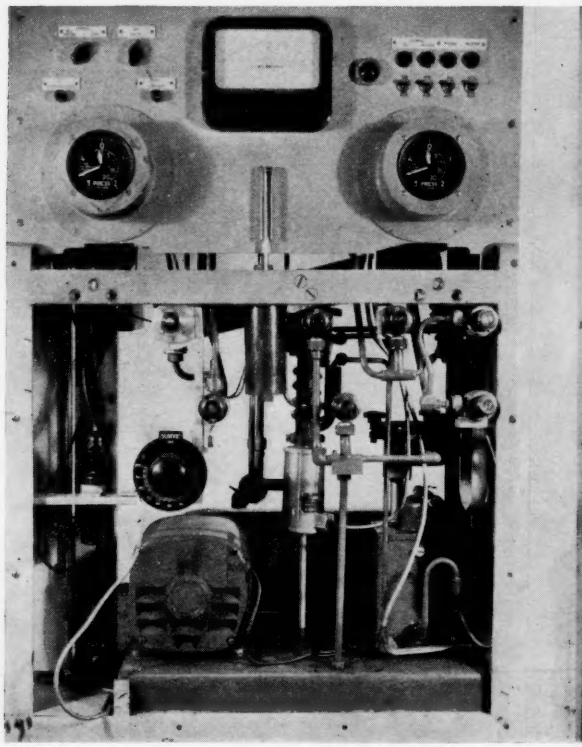

Fig. 2.

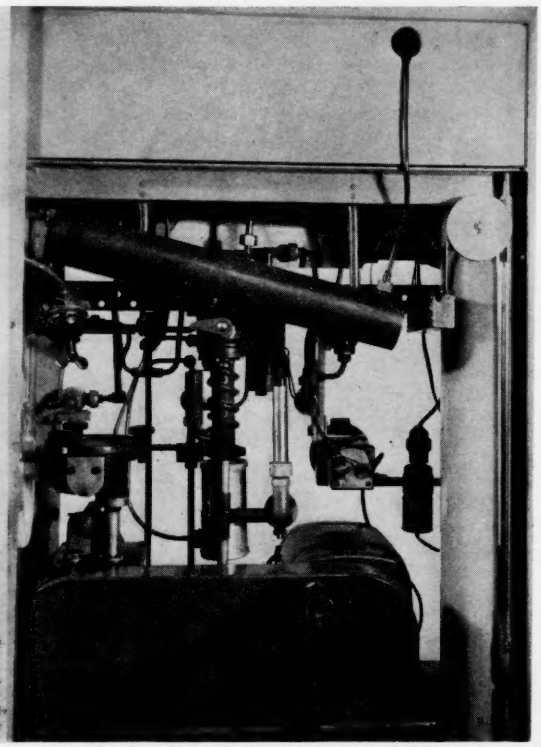

Fig. 2a.

Front view and rear view respectively of the all-metal apparatus.

The Vacuum System

This is a commercial pumping unit consisting of a rotary oil pump and a 1-inch oil diffusion pump, all mounted on a baseplate complete with pump cooling water inlet and outlet connections, vacuum release,

pump by-pass, isolating and baffle valves. The main modifications needed are an extension spindle to the vacuum release valve, and a rotary toggle extension inserted in the diffusion pump heater leads. This switch is closed by the expansion of a metal bellows connected to the inlet water supply, and ensures that

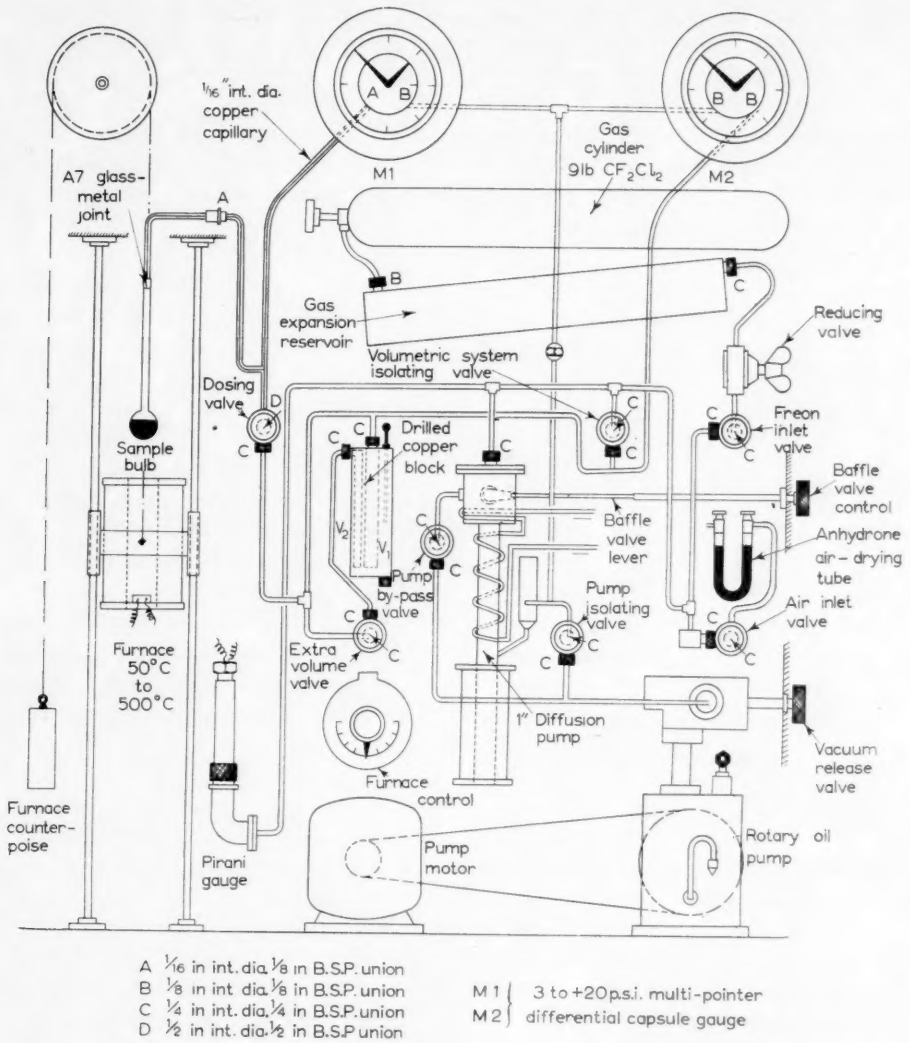

Fig. 3. Diagramatic view of the layout of the apparatus seen from the front.

to the baffle valve lever. Backing vacuum for the differential capsule gauges is obtained by tee-ing into the low vacuum line between the diffusion pump and the pump isolation valve. A micro-switch is the heaters cannot be switched on before the cooling water is turned on.

The degree of vacuum attained is measured by a commercial metal Pirani gauge head connected to

the diffusion pump head. The method of wiring shown in Fig. 4 enables the gauge to be used as a leak detector and as a degassing indicator. If the meter is adjusted to centre zero by the *vac. set* control, with both pumps running in the closed leak-tight system, then this same zero reading will indicate a satisfactory degassed state of the sample in subsequent experiments. The *at. set* control is adjusted similarly, but at atmospheric pressure; in this condition the gauge functions as a leak-detector, using a 'hydrogen-probe'. As an alternative to this bridge system, a commercial Pirani gauge may be used as a separate item.

Calibration

This is best effected by connecting glass bulbs of known volume fitted with stopcocks and A7 sockets,

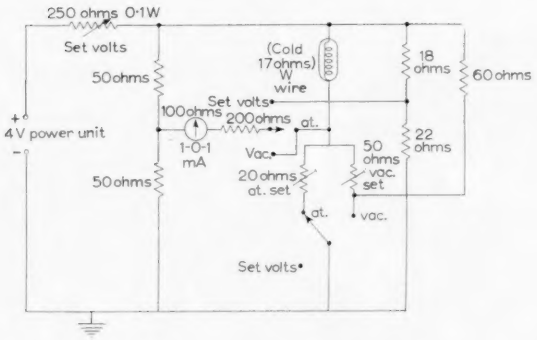

Fig. 4. Circuit for Pirani gauge.

in place of the sample container of the adsorption system. The recommended procedure is as follows:

(i) Connect the bulb to the A7 cone with the stopcock open.

(ii) Evacuate the apparatus and the bulb, then close the glass stopcock.

(iii) Admit air up to 20 p.s.i. (P_1), then close the dosing valve.

(iv) Open the glass stopcock and note the new pressure reading, (P_2).

This procedure should be repeated using the same bulb but starting at different values of P_1.

Recommended values for the volumes of the bulbs are 10, 20 and 150 c.c.

The volume of the dosing system is then determined in a similar manner using the combined volumes of the calibrated bulb and the adsorption system. As a final check, the total volume of the adsorption and dosing systems should be determined using the 150 c.c. capacity bulb. It is important to note that in any experiment involving the dosing valve, the final readings must be taken with this valve shut, owing to the change in volume which takes place as the valve mechanism is operated.

Errors and Corrections

The type of gauge used in this equipment is calibrated in 0.05 p.s.i. divisions (about 2.5 mm.Hg per division) and the tenth sub-division of these can be estimated by eye. The sticking error of the mechanism is about 0.01 p.s.i. lag on the reading, but the combined backlash and mechanical faults may amount to $\pm$0.05 p.s.i. The gauge manufacturers supply correction charts for the mechanical faults, corrected to the nearest 0.01 p.s.i., and in practice the final corrected readings have been found to be sufficiently accurate for all the purposes for which this adsorption apparatus is intended.

Although the movement of the pressure-sensitive capsule is very small, there is nevertheless a volume change, roughly linear with pressure, amounting to about 3 c.c. when the pressure is increased from 0 to 20 p.s.i. The effect can be measured from the curvature of the plot P_1 against P_2 obtained in the calibration of the adsorption system. For routine work it is possible to ignore the curvature and to calculate an average volume by drawing the best straight line through the curve and the origin. Otherwise, a factor of proportionality between the volume expansion and the pressure should be calculated and used to obtain the true volume of the system corresponding to every pressure reading. However, it will usually be found after a few trial experiments that satisfactory results can be obtained by taking average values of the gauge volumes over the pressure ranges 0–4 p.s.i., and 10–20 p.s.i. for the gauges in the adsorption and dosing systems respectively.

OPERATION OF THE APPARATUS

Sequence of Operations

The following is a detailed sequence of the operation of the apparatus:—

(1) Three-quarter fill the sample bulb with the powder under test, insert a plug of glass wool in the tube and push it down with the filling rod. Grease the A7 metal cone and connect the bulb to the apparatus by the A7 glass socket.

(2) Shut valve V7 and start the rotary pump, open V5 slowly to avoid unnecessary strain on the differential gauge mechanism, turn on the cooling water and switch on the diffusion pump. Open valves V4 and V2. As soon as gauge E shows zero pressure, open the dosing valve D very cautiously, watching the sample to see that it does not fluidise in the bulb. When gauges C and E both show zero pressure, open D to its fullest extent, shut valves V2 and V4 and open the baffle valve V6. Adjust the Pirani on *vac. set* to read zero and re-open V2.

(3) Set the furnace regulator to give the desired degassing temperature and raise the furnace up round the bulb. A light metal disc slipped over the A7 socket is often useful to protect the greased joint. The heater may now be kept in position for a predetermined length of time for routine determination, or allowed to remain until the Pirani meter has again fallen to zero if the degassing is desired to be as complete as possible.

(4) When degassing is complete remove the heater, shut valves D and V6, and carefully open V1 to admit air from a cylinder. Nitrogen or helium, depending on which gas has been found to be least adsorbed on the particular material under test at the temperature of the particular refrigerant bath in use, may be admitted instead of air, if desired.

(5) When gauge E reads about 15 p.s.i., shut V1 and V2, and open V4. Read gauge E accurately.

(6) Bring the refrigerant bath up round the sample bath, and cautiously admit a small volume of gas from the dosing system into the adsorption system. Shut D and wait for the reading on C to become steady. Read gauges C and E.

(7) Repeat the dosing procedure a number of times until the final steady pressure on C is about 0.35 P_o, where P_o is the saturation vapour pressure of the adsorbate gas at the temperature of the refrigerant bath. A list of suitable adsorbate gases and baths is given in the previous paper.

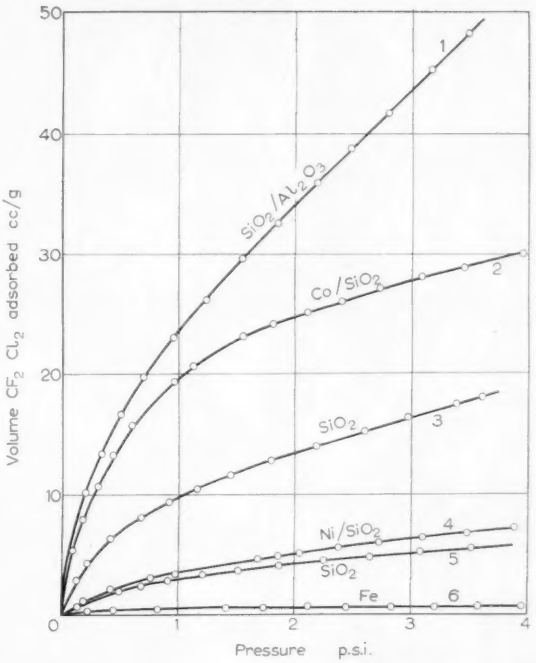

Fig. 5. Adsorption isotherms of CF_2Cl_2 on various adsorbents.

(8) Open V2 and D in that order, remove the bath and replace the furnace. When the pressure on gauge C has fallen to zero, shut V4 and open V6.

(9) Repeat the operations (4) to (7) using Freon (or other adsorbate gas) admitted through valve V3.

(10) Close down by admitting air to the gauges *via* valves V1 and V2 and then reversing operations (1) and (2).

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The required final data are the volumes of gas adsorbed at the corresponding equilibrium pressures. These values are simply obtained by first applying the corrections to the gauge readings, and then using the known volume of the dosing system and the readings of gauge E to calculate the volume of gas admitted. It should be noted that using this double gauge system the difference between the *initial* '*E*' gauge reading *and* any subsequent 'E' gauge reading gives the *total* volume admitted up to that time. This

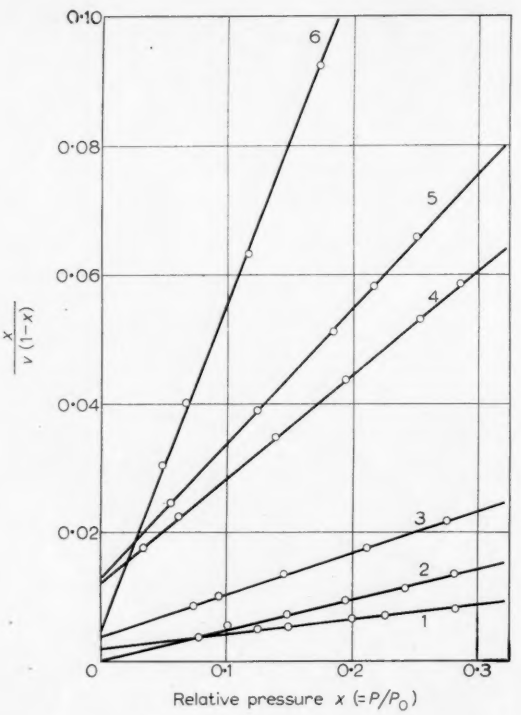

Fig. 6. B.E.T. plots of adsorption of CF_2Cl_2 on various adsorbents.

eliminates the errors due to the summation of a number of small doses which often occur when using an apparatus in which a single gauge or manometer serves to indicate the pressure in both the dosing and adsorption systems.

Details of the method of calculating the surface area from the adsorption data are given in the previous paper and the references tabulated therein.

Single-Point 'Surface Area' Determinations

For the routine comparison of batches of the same material it is often sufficient to compare the volumes of gas adsorbed, by the same weight of different samples at the same pressure. For this purpose it is even unnecessary to determine the dead space. A simple procedure using the present apparatus is as follows:—

(*a*) Weigh the same amount of sample into standard size sample bulbs for each determination.
(*b*) Degas under identical conditions.
(*c*) Fill the dosing system up to the same pressure in every determination.
(*d*) Open 'D' and read the final pressure.

By using different weights of the same sample (correcting if necessary for the difference in volume occupied by the sample) it is possible to construct a chart from which the specific surface of any other sample of similar material can be read off from the value of the final pressure obtained in (d) above.

RESULTS OBTAINED WITH THE APPARATUS

Figs. 5 and 6 are, respectively, the adsorption isotherms and the B.E.T. plots of the adsorption of dichlorodifluoromethane at −33.1°C on a number of adsorbents, of widely differing B.E.T. 'C' values and surface areas obtained with the present apparatus. For Curve 6, Fig. 6, the ordinate has been divided by two in order to show enough points on the same graph. Data relating to these graphs is given in Table 1, in which the time includes the times taken for degassing and for calculation of the results.

The lower pumping speed at the sample bulb in the metal apparatus is largely responsible for the smaller surface areas, since over the same period it results in inadequate degassing of the sample. The smaller absolute values are actually immaterial because adherence to a fixed degassing routine affects all samples more or less equally as shown by the good agreement between the values of the relative specific surface.

DETERMINATION OF SORPTION RATES

These can be determined by following the procedure already given for the determination of specific surface, up to the point at which the first dose is to be admitted. At this point the dosing valve is opened rapidly at the same time as a stopwatch is started. The readings of the gauge are then taken starting at intervals of one second and lengthening the interval as the rate of sorption slows down. This method has proved very satisfactory for obtaining rate curves for the rate of adsorption of argon, methane and nitrogen by anthracite at temperatures between −78 and +30°C.

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Table I. **Comparison of Methods and Apparatus.**

Adsorbent (See Fig.5)	*Apparatus*	*Method*	*Time Taken Hours*	*Specific Surface m^2g*	*Relative* Specific Surface*
(1) SiO_2	Glass	B.E.T.	4	564	1.0
	Metal	,,	1½	470	1.0
	,,	Single Point	¾	442	1.0
(2)Co/SiO_2	Glass	B.E.T.	2	242	0.429
	Metal	,,	1	237	0.504
	,,	Single Point	½	299	0.677
(3) SiO_2	Glass	B.E.T.	4	198	0.351
	Metal	,,	1½	163	0.347
	,,	Single Point	¾	168	0.380
(4)Ni/SiO	Glass	B.E.T.	4	81	0.144
	Metal	,,	1½	65	0.138
	,,	Single Point	¾	63	0.143
(5)SiO_2	Glass	B.E.T.	4	60	0.106
	Metal	,,	1½	50.5	0.107
	,,	Single Point	¾	52	0.118
(6) Fe	Glass	B.E.T.	2	4.85	0.0086
	Metal	,,	1	4.45	0.0095
	,,	Single Point	½	5.44	0.0124

* Relative to the values of the specific surface of sample (1) as determined by each of the three different methods.

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THE PURIFICATION OF BERYLLIUM by distillation

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Summary

THE PURIFICATION OF BERYLLIUM by distillation is considered and a quantitative evaluation made on the basis of the relative vapour pressures of the major impurities present. An appraisal is made of the practical considerations governing fractional distillation on a laboratory scale.

Sommaire

ON CONSIDERE la purification du beryllium par distillation, et une évaluation quantitative basée sur les tensions de vapeur relatives des principales impuretés présentes. On évalue les considérations pratiques gouvernant la distillation fractionnante à l'échelle de laboratoire.

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INTRODUCTION

BERYLLIUM METAL HAS nuclear properties which, in any normal metal, would ensure its place at the fore of the ever developing atomic industry. Among these properties are its low neutron-capture cross section, its high neutron reflectivity and its high moderating power for neutrons. Moreover, as a canning material for fuel rods for thermal reactors it is unsurpassed in its nuclear properties by any other metal or alloy. The main deterrent to its widespread use is that, in most metallurgical conditions, it is almost completely brittle at room temperature.

Two main theories have been advanced to account for the lack of ductility shown by even the purest forms of beryllium metal obtained to date. The first considers the crystal structure of the hexagonal close-packed metal, suggesting that the lattice geometry and other characteristics do not favour deformation by slip. The second theory embraces the probable effect of small amounts of impurity in the lattice. It now appears probable that both factors contribute to the inductility of beryllium, and that if the metal could be produced with no impurity content, a limited but definite ductility would be found, such as exists in similar metals such as titanium and zirconium. As yet, metal sufficiently pure to demonstrate ductility has not been made; it is hoped that purification by distillation may produce it.

*MS. received December, 1956.

HISTORICAL REVIEW

Sloman, in 1932, published the first account of a process involving the distillation of beryllium[1]. A high frequency furnace was used, in which the beryllium, held in a beryllia crucible was heated, by induction, to a temperature of 1,850°C–1,900°C, under a pressure of 0.005 mm. Hg. A small amount of distillate was recovered, in the form of sheet, 1 mm. thick, which was found to have a Brinell hardness of 60–65, the lowest figure to be reported, even in more recent researches. Although no tests of ductility could be carried out, the author reported that the sheet could be flexed without immediate fracture.

Kroll,[2,3] in 1935, improved on Sloman's apparatus by using an all-metal/quartz device, eliminating

rubber plugs and pipes, but depending still on the use of Apiezon greases for sealing. A 40 g. charge was heated to 1,400°C, and a 15 g. distillate obtained, in which the iron and silicon had been greatly reduced below their original contents. Aluminium, magnesium and manganese were, however, increased in amount. No substantial hot malleability was found in the distilled material.

Claussen and Skehan[4] recorded attempts to purify beryllium in 1942, but reported that little purification was, in fact, achieved on vacuum-melted 99.9% pure metal, apart from a reduction of the aluminium content to less than 0.001%. Small crystals of distillate were found to hot-roll well, but no ductility was found in the larger pieces of distilled material.

Metal of 99.962% purity was reported to have been produced by Losano, but no mechanical tests were carried out.[5] Other tests such as electrical conductivity measurements, indicated that such physical properties altered very rapidly as the metal purity exceeded 99.6%. No mechanical tests were reported in the researches of Bradner[6] or Smith[7] either, who produced thin sheets of beryllium by distillation, for use as x-ray windows. Bradner reported, however, that even such thin foils, 200 micro-inches thick, were very brittle. Smith, who evaporated beryllium held on a graphite support, using an electron bombardment technique, maintained that the sheets, 0.16 mm. thick, were malleable, but gave no supporting evidence. X-ray powder photographs failed to detect any structural differences between the metal before and after evaporation.

The latest large-scale research to be published was that carried out at Massachusetts Institute of Technology under the auspices of the U.S. Atomic Anergy Commission. Kaufmann[8] reported that distillation was accomplished on a 6-inch. diameter crucible scale, in a vacuum of 10^{-4}mm. Hg. The aluminium, silicon and oxygen contents of the distillate were found to be unaltered by the process, but considerable purifications resulted with respect to iron, carbon and boron. No great ductility was found even with individual small crystals of the condensate. On a smaller scale however, the most successful attempt to reduce the oxygen and nitrogen contents of beryllium was made by Pearsall in 1952[9]. Several distillations were carried out in a vacuum of the order 10^{-8}–10^{-9}mm. Hg using a glass container capable of being outgassed at 450°C, and furnace parts of tantalum which were outgassed at about 2,000°C. Only 10 g. of metal were distilled, and a 0.010–0.040 inch thick condensate recovered, which was substantially lower in copper, magnesium, silicon and oxygen, but greatly enriched in chromium and iron, with calcium, manganese, nickel and chlorine relatively unaffected. No ductility was observed in the metal as deposited.

Apart from this latter research, however, no genuine attempt has been made to eliminate oxygen, nitrogen or carbon contamination of the metal condensate. The outgassing of surfaces within the vacuum chamber has either not been attempted, or has been rendered impracticable by the unrestricted use of porous refractories. With the exception of Pearsall's work, in-leakage of oxygen and nitrogen has in all cases been allowed, and the use of vacuum sealing waxes and oil diffusion pumps has necessarily resulted in the direct access of carbonaceous materials to the vacuum chamber. Even so, some fractional distillation, particularly in Kaufmann's work, resulted in a reduction of metal impurity content in the condensate, whereas in Pearsall's apparatus little reduction in metallics was obtained.

In addition, all previous investigators have failed to produce from the recovered condensate, a metal in a state metallurgically suitable for mechanical testing. Bending tests on thin sheets of condensed metal are open to considerable criticism, and no valid conclusions as to the true ductility of the metal can be made from such tests. Such amalgamation as has been used in some of the above researches, moreover, may well have introduced contamination.

It may be held, therefore, that the experiments conducted to date have not produced as pure a metal as may possibly be obtained, nor investigated sufficiently the mechanical properties of the distillate.

THEORETICAL CONSIDERATIONS

It is necessary to estimate the probable effects

of time and temperature on the course of distillation. Two basic assumptions must be made:

(*a*) That the rate of evaporation of a metal is directly proportional to its vapour pressure. (An assumption developed from the basic gas laws and evaluated by Langmuir[10])

(*b*) That a metal dissolved in solid solution exerts a vapour pressure proportional to its molecular concentration (Raoult's Law).

Using neither of the above considerations, but from a purely mathematical basis Rayleigh developed an equation for which a graphical solution must be sought:—

$$Log_e \frac{W_0}{W_1} = \int_{x_t}^{x_o} \frac{dx}{y - x,}$$

where W_o = Initial weight of melt,
x = Percentage of alloying constituent in melt,
y = Percentage of this constituent in the vapour phase.

Betcherman and Pidgeon[11] attempted graphical analysis for the distillation of a magnesium-calcium alloy and concluded that such calculations could not be used to yield quantitative results.

The Rayleigh equation may be developed further, however, if one assumes that Raoult's law applies, that is that the partial pressure of each constituent in the vapour is equal to the product of the vapour pressure of the pure constituent and its molar concentrations in the solution. Then, if the original melt contains N_a moles of A, and N_b moles of B, after a short time of distillation, $N_a - dN_a$ moles of A and $N_b - dN_b$ moles of B remain.

That is, dN_a moles of A have evaporated. This is equal to

(the molar rate of evaporation of A) × (the molar fraction of A in the melt) × time $= R_a \cdot \frac{N_a}{N_a + N_b} \cdot dt$ (1)

Similarly,

$$dN_b = R_b \cdot \frac{N_b}{N_a + N_b} \cdot dt \qquad (2)$$

Dividing (1) by (2)

$$\frac{dN_a}{dN_b} = \frac{R_a \cdot \frac{N_a}{N_a + N_b} \cdot dt}{R_b \cdot \frac{N_b}{N_a + N_b} \cdot dt}$$

$$= \frac{R_a \cdot N_a}{R_b \cdot N_b} \qquad (3)$$

Thus,

$$\frac{dN_b}{N_b} = \frac{R_b}{R_a} \cdot \frac{dN_a,}{N_a}$$

and, since R_a and R_b are constants at a given temperature,

$$\int_{N_b\,(o)}^{N_b} \frac{dN_b}{N_b} = \frac{R_b}{R_a} \int_{N_a\,(o)}^{N_a} \frac{dN_a}{N_a}$$

Integrating,

$$Log_e \frac{N_b}{N_{b(o)}} = \frac{R_b}{R_a} log_e \frac{N_a}{N_{a(o)}} \qquad (4)$$

To solve Equ. 4 in terms of percentage by weight:

If x_o is the original percentage by weight of A in a melt of total weight W_o, and x_1 is the final percentage in the remaining melt of weight W_1, then

$$N_{a(o)} = \frac{W_o\, x_o,}{100 M_a}$$

where M_a is the molecular weight of A.

Also

$$N_{b(o)} = \frac{W_o\,(100 - x_o)}{100 M_b};$$

$$N_a = \frac{W_1\ x_1}{100 M_a}$$

and

$$N_b = \frac{W_1\,(100 - x_1)}{100 M_b}$$

Therefore, substituting for N_a and N_b in Equ. 4 since

$$log \frac{N_b}{N_{b(o)}} = \frac{R_b}{R_a} log \frac{N_a}{N_{a(o)}},$$

$$log \frac{\frac{W_1 (100 - x_1)}{100M_b}}{\frac{W_o (100 - x_o)}{100M_b}} = \frac{R_b}{R_a} log \frac{\frac{W_1 x_1}{100M_a}}{\frac{W_o x_o}{100M_a}}$$

$$i.e. \quad log \frac{W_1}{W_o} \cdot \frac{(100 - x_1)}{(100 - x_o)} = \frac{R_b}{R_a} log \frac{W_1}{W_o} \cdot \frac{x_1}{x_o}$$

$$i.e. \quad log \frac{W_1}{W_o} = \frac{R_a}{R_a - R_b} \left(\frac{R_b}{R_a} log \frac{x_1}{x_o} - log \frac{(100 - x_1)}{(100 - x_o)} \right) \quad (5)$$

The molar rate of evaporation R, is connected to the vapour pressure by the relation[12]:—

$$R = \frac{5.833 \times 10^{-2} P}{\sqrt{MT}} \quad moles/cm^2/sec.$$

$$\therefore \frac{R_a}{R_b} = \frac{P_a}{P_b} \sqrt{\frac{M_b}{M_a}}$$

where

P = Vapour pressure in millimetres Hg
M = Molecular weight
T = Temperature in degrees Kelvin.

Evaluation of Distillation Equations in Beryllium Purification

It is possible to calculate, from Equ. 5, the impurity content of the unevaporated melt during the course of distillation. The simplest way is to assume the original weight of melt to be 100 g.; that this contained x_o g. of a given impurity, and that after a certain time of distillation x_1% remains in the melt. If simple ratios of x_1/x_o be taken, such as 10, 100, 1,000, the ratios W_1/W_o are readily calculated. (If the impurity element has a higher vapour pressure than the solute element, diminishing ratios such as 1/10, 1/100, etc. must be taken).

From these figures the amount of distillate, and its impurity content may be calculated. It has been found essential, in calculations involving even small values of x_1, that the factor

$$log \frac{100 - x_1}{100 - x_o}$$

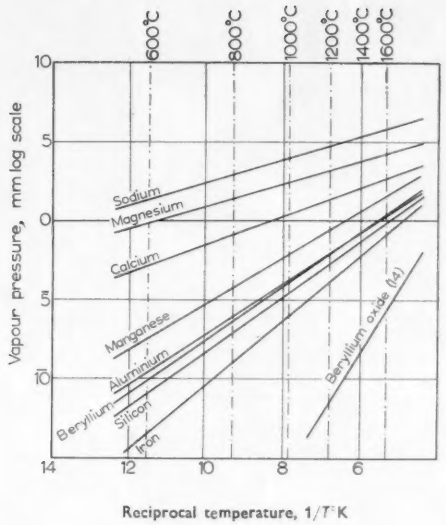

Fig. 1. Vapour pressures of pure metals. (Collated by Kubaschewski and Evans.)

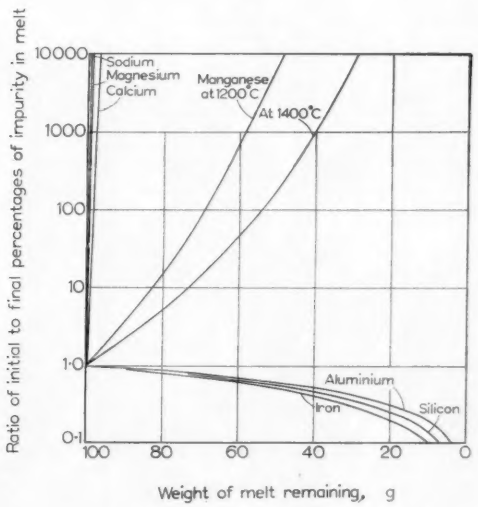

Fig. 2. Change in melt composition during distillation at 1400° C.

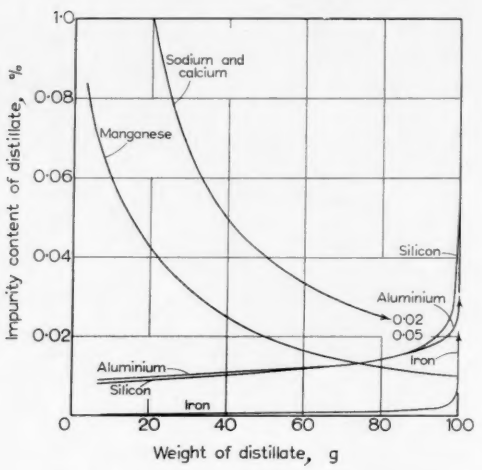

Fig. 3. Change in condensate composition during distillation at 1400°C.

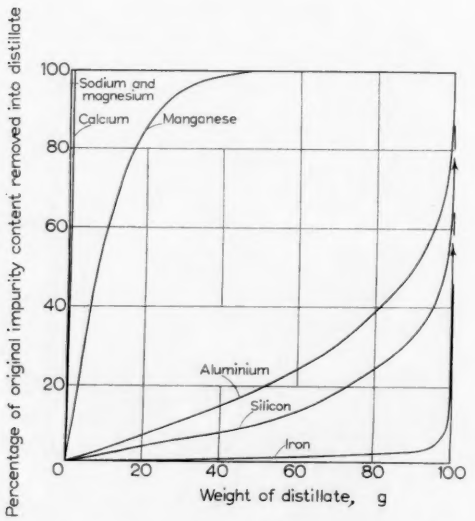

Fig. 4. Percentage of impurity removed during distillation at 1400°C.

should not be neglected, as this can lead to completely erroneous results.

To calculate the distillate composition to be obtained under a given set of experimental conditions, the composition of the starting material must be known. For the purposes of the present investigation a composition has been assumed, based on the normal impurity contents found in commercial French Flake or Brush Bar Beryllium; this is

0.02% Na, 0.03% Mg, 0.02% Ca, 0.01% Mn, 0.05% Fe, 0.4% Be0 0.03% Al, 0.05% Si,

The values taken for the vapour pressures of these metals are those collated by Kubaschewski[13], and presented in Fig. 1. Considerable doubt still exists about the accuracy of the vapour pressure data available, especially with respect to those metals which form highly tenacious surface films, *e.g.* aluminium and beryllium. For this latter metal, the values taken are those given by Holden, Speiser and Johnston[14] which has been confirmed by Gulbransen[15].

The calculated results are presented graphically in Figs. 2, 3 and 4. Fig. 2 indicates the change in composition of the melt during distillation. Fig. 3 shows the changing composition of the distillate. The amount of impurity finding its way into the distillate is then given in Fig. 4, being expressed as a percentage with the original content as 100%.

Conclusions. From the above the following can be concluded:—

(1) All calcium, sodium and magnesium should be completely removed with the first two grams of distillate. Manganese may be removed more slowly, but should be 90% evaporated (leaving 0.001 g. in the melt) in the first 24 g.

(2) Aluminium and silicon are difficult to remove since they evaporate at a rate comparable to that of beryllium. If the first 20 g. of distillate are rejected and distillation discontinued after a further 50 g. have evaporated, this 50 g. will still contain 23% of the original aluminium, and 18% of the silicon—a content of 0.0138% and 0.018% respectively.

Effect of Oxide Crucible and Oxide Content of Distillate

From the vapour pressure data on beryllia given by Erway and Seifert[16] the rate of evaporation at 1,500°C can be calculated to be 1.172×10^{-8} g./cm^2/

sec. With a crucible of 3 inch. diameter and 7 inch. high, the exposed surface area will give a rate of evaporation of 5.92×10^{-5} g./minute.

The rate of evaporation of beryllium from a 3-inch. diameter orifice at 1,400°C is 2.39 g./minute. The oxide content should, therefore, be 0.0025%. Reducing this figure may necessitate operating at a lower temperature, or the use of a different refractory container, such as tantalum.

It is obvious that the rate of evaporation of the beryllia in solid solution is negligibly small compared with that of the slowest-removed metallic impurity, since its vapour pressure is 1,000,000 times less than that of iron. If, therefore, a suitable refractory container other than beryllia can be used no oxide contamination due to evaporation would be found. Thermodynamic data also indicates that negligible decomposition of beryllia should occur.

Effect of the Presence of Gas Molecules Above the Melt

The foregoing theory assumes the complete absence of gas molecules above the melt; for practical purposes this simplification may give a sufficiently accurate picture provided that the pressure inside the chamber is of the order of 10^{-3} mm. Inaccuracies arise, however, when the pressure of the inert gas is higher, or when a high rate of evaporation is attempted. The rate of distillation is dependent on the difference in pressure at the condenser surface and the evaporating surface, *i.e.* distillation is dependent on diffusion of the metal molecules across this concentration gradient. The greater the metal 'gas' concentration at the melt surface, however, the slower is the net rate of exodus of molecules from the surface.

Various attempts have been made to interpret, mathematically, the effect of gas pressure above the melt surface[17], but it appears that, only when the pressure is appreciably greater or less than the vapour pressure of the melt, is the analysis successful. However, it has been shown practically, that regulation of an inert gas pressure can be used with advantage to alter the ratio of the rates of evaporation of two metals in solid solution, and that empirical methods of determining the optimum pressure conditions are to be preferred to mathematical analyses.

Regulation of the argon pressure in the vacuum chamber may afford a means of increasing the purity of the recovered beryllium distillate, or of increasing the yield of metal of a given purity.

The form of distillate is also affected by the gas pressure in the chamber. Barton found that, during calcium distillation at low pressures, a tough compact and strongly adherent condensate was formed, whereas at higher pressures the crystals became smaller and more friable and could be broken in a ball mill.

PRACTICAL CONSIDERATIONS

Apparatus has now been built, in which it is hoped to verify the foregoing theoretical calculations, and in which beryllium may be prepared in a greater state of purity than has hitherto been possible. In order that sufficient material should be prepared to enable mechanical test specimens to be made the apparatus has been designed to hold a 500 g. charge. This is contained in a beryllia crucible, which is surrounded by a cylindrical tantalum susceptor and tantalum radiation shields inside a high frequency coil. The condenser is a flat reversible plate held horizontally above the crucible. The distance between the plate and the top of the crucible can be altered, so that the condenser temperature attained by radiation from the furnace can be altered within certain limits. In addition, the condenser can be water-cooled if desired, and, in its top position, can be turned over during the distillation run, by remote control from outside the vacuum system. In this way the distillate can be divided into two parts, the first high in the volatile elements, sodium, calcium, magnesium and manganese; and the second low in these. Distillation will be discontinued before the bulk of the less volatile constituents, aluminium, silicon, and iron have evaporated to any great extent.

The furnace and condenser are contained in a larger water-cooled, stainless steel bell, which rests on a stainless steel base plate. To avoid ingress of carbonaceous materials, oxygen, nitrogen, or other

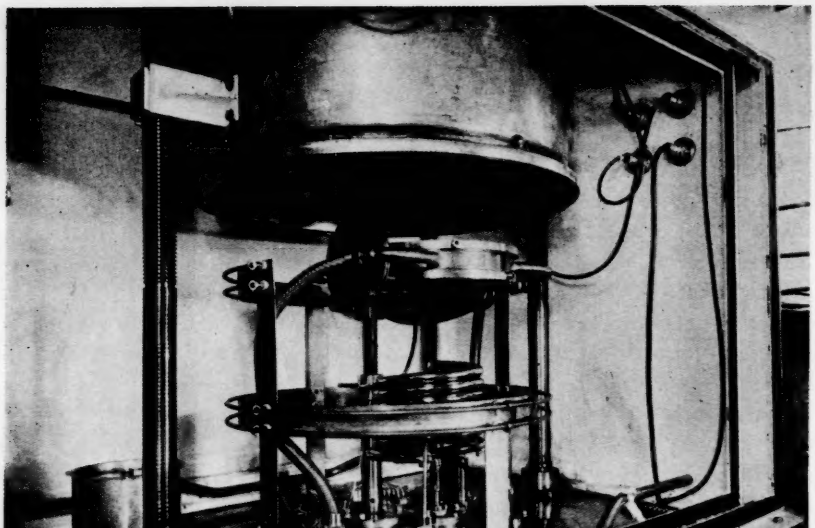

Fig. 5. Furnace assembly.

undesirable contaminants the following precautions have been observed:—

(*a*) The system is evacuated by a mercury diffusion pump, with an auxiliary liquid nitrogen trap. Contamination by diffusion pump oil, even when a cold-trap is fitted has been noticed in other investigations.

(*b*) No rubber materials or vacuum greases or oils are present in the vacuum chamber. Throughout the whole of the high vacuum system the seals are of sheet aluminium for the permanent joints and indium wire for the readily demountable joints, such as between the bottom of the bell and the base plate.

(*c*) Outgassing of the bell walls can be achieved by glow discharge. High voltage a.c. is imposed on two stainless steel rods that encircle the furnace, inside the vacuum system, within an inch or so of the bell walls. If a dynamic argon flow is established through the system at about 90–100 μ pressure, a glow discharge can be initiated with 1,000–2,000 V potential applied to the electrodes. With a suitable geometry the glow discharge fills the vacuum chamber.

(*d*) The entire vacuum system, with the exception of the backing line and rotor pump is enclosed in an argon shroud, filled with calcium-purified argon. Any accidental inleakage into the system during operation or shut-down is pure argon. Handling of materials inside the chamber is by means of rubber gloves, and materials may be introduced into the argon shroud by means of an evacuated transit tunnel. Contact with air is thereby minimised before, during and after the distillation run, and the distillate need never come in contact with air during the stripping operation or during the subsequent agglomeration techniques.

A photograph of the apparatus, revealing the main points mentioned above, is shown in Fig. 5. For the sake of clarity the crucible and its supports have been

omitted, as has the front Perspex panel on the argon shroud.

CONCLUSIONS

An evaluation has been made of the possibility of obtaining purified beryllium metal by distillation. From theoretical principles it appears that a batch distillation process must be divided into three parts, in which the first portions of distillate are discarded together with the last portions remaining in the crucible. Precautions to avoid contamination of the product must be exceptionally stringent and require refined vacuum techniques.

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THE ADHESION OF ALUMINIUM FILMS ON GLASS AFTER IONIC BOMBARDMENT

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Summary

EXPERIMENTS WERE PERFORMED with the purpose of explaining both the mechanism of the process called cleaning by ion bombardment, applied to glass surfaces prior to the deposition of films, and of determining the conditions for carrying out such a process in order to secure good film adhesion on the supporting surface. As a test for film adhesion, the process of anodising was used. Three main experimental observations are recorded: (1) The presence of an adsorbed skin of molecules on the target surface; (2) the instant appearance of the effect; and (3) the destruction of this effect by rubbing. These confirm the suggestion that the high adhesion of the deposit following the electrical treatment is due to the creation of numerous centres of condensation by electrically charged gas molecules adsorbed on the surface. With regard to the practical usefulness of the process, the optimum values of various factors which influence the process are given.

Sommaire

DES RECHERCHES furent faites dans le but d'expliquer les deux mécanismes du procédé appelé 'nettoyage par bombardment ionique,' appliqué aux surfaces de verre, d'abord pour la déposition de films, et ensuite pour déterminer les conditions les meilleures pour une bonne adhésion sur la surface du support. Pour essayer l'adhésion des films, le procédé electrolytique, fut employé. Trois observations expérimentales principales, one été notées: (1) La présence d'une couche adsorbée de molécules sur la surface de la cible; (2) L'apparition immédiate de l'éffet, et (3) La destruction de cet effet par frottement. Ces données confirment la suggestion que la forte adhésion du dépôt aprés le traitement électrique est due à la création de nombreux centres de condensation formés par des molécules de gaz chargées électriquement et adsorbées sur la surface. Les valeurs optima des différents facteurs qui influencent ce procédé sont données tout en se référant à son utilité pratique.

INTRODUCTION

INCREASING APPLICATION of thin evaporated films, metallic and dielectric, have occasioned numerous investigations into the conditions of their production in order to obtain acceptable results. However, there are only a few papers specifying the factors influencing the basic requirements of a coating, *i.e.* stability and adhesion to its support. The problem of the cleanliness of the substrate, onto which the material condenses, has never been investigated methodically. Thus, almost all statements on the preparation of the target surface are lacking the desired accuracy, or misleading.

Practice has shown that the stability and adhesion of a film are still poor,† even if (*a*) the surface to be coated is free of any visible impurities after thorough cleaning by washing, (*b*) the best vacuum conditions are applied, and (*c*) the evaporation is carried out either at a high or a low rate.

This was the position two decades ago, when the importance of thin films in research and applications became apparent.

Cartwright[2] had already suggested in 1930 a method for increasing the adhesion of sputtered films by heating the support in vacuum up to 200°C. However, this procedure did not give the expected results, particularly when applied to evaporated films.

* MS. received November, 1956.

† In very unfavourable conditions: films with blisters may even be obtained.[1]

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It was not until 1934 that Strong[3], following a conclusion by Baker[4], introduced into practice the treatment of the supporting surfaces by an electrical glow discharge. The efficacy of this method has been proved by its extensive use in the production of many kinds of films. As the matter stands to-day we may well assume that the decisive factor in the stability and adhesion of a film is the electrical treatment of the supporting surface, prior to the evaporation process. In particular, the effect of this process on the stability of aluminium films can be easily shown: a non-anodised aluminium film remains clear on the treated portion of the target indefinitely, but the imperfections (smears) remaining after physical cleaning are still visible on the untreated portion.

In an attempt to explain the 'breath figures' produced by a flame or by a glow discharge, Baker[4] assumed that their origin is to be attributed in part to the burning-off or volatilisation of a thin film of contamination allegedly still present on a previously washed surface. Therefore, as a result of this action, the surface is indeed uncontaminated, and moisture condenses on it in the form of a continuous transparent film. Baker noticed also the extraordinary persistence of this cleaning action.

Strong[5] continued the investigations, and concluded that the breath figures are due to 'the removal of some substance, probably a fatty acid, from the surface by ionic bombardment'.

Lack of conclusive experimental results on the effects of the glow discharge has led to further assumptions made to explain the nature of the process: (*1*) The target's surface is brought to a high temperature by the impact of the positive ions accelerated by the applied electric field. The process, therefore, would consist in an energetic degassing of the surface[6]. Hiesinger[7] states: 'In order to obtain a good adhesion of the film, the glow discharge should be carried on until the heating reaches the upper limit permissible for the target'. (*2*) The bombarding positive ions remove any layers of gases and water adsorbed on the surface after the preliminary cleaning process[8]. (3) Chemical reactions take place between adsorbed impurities and active gases in the glow discharge[8a]. (4) The ions hitting the surface produce condensation or crystallisation nuclei necessary for good adhesion of the film deposit[9].

None of the investigations published so far yielded results properly establishing which hypothesis is correct. Our own experimental results contradict most conclusions so far reached on the mechanism of the process of electric treatment as a method of preparing surfaces for adherent depositions.

Although in numerous papers emphasis is placed on the necessity of using this 'electrical cleaning' or 'ion bombardment' in obtaining films of good quality, nevertheless the method is considered merely as a practical process evolved empirically and having no particular scientific significance. At the Symposium on Thin Films[10] held in 1951, the electric treatment was not mentioned in the pronouncements on adhesion between a solid substrate and a deposited film.

The physical nature of this process being unknown, the conditions for carrying it out can be given only approximately. The experimental procedure normally employed at present[8] is as follows: After scrupulous physical cleaning, the target is placed in the evaporation chamber. At a relatively low pressure (normally of air) an electrical discharge is initiated, and continued for a while, such that the glow covers the surface.

Summing up, the values of various factors of the experimental procedure given in the scientific and technical literature are the following:

Voltage of the source:	from 700 to 30,000 V.
Discharge current:	from 15 to 600 mA.
Pressure in work chamber:	from 0.4 to 10^{-4}mm.Hg.
Time of discharge:	from 10 to 30 minutes.

d.c. discharge in air is mostly used.

Of course, these values also depend on the size of the evaporation system. However, these wide ranges show that knowledge on electrical 'cleaning' is rudimentary at present, and any optimum conditions cannot be properly selected, as some authors claimed[11]. In practice, any 'excess' treatment should be avoided to minimise costs. A systematical investigation into this problem was, therefore, desirable.

METHOD OF TESTING ADHESION

It has been shown experimentally[12] that the coefficient of sliding friction of dry surfaces of metals and glass considerably increases when these surfaces are heated in a vacuum. Initially this result was explained by postulating the removal of adsorbed layers or impurities[13], and later it was shown[14] that the effect is due to degassing of the surface. As such surfaces are generally called 'clean' surfaces, it follows that the coefficient of friction should be an accurate measure of the cleanliness of the surface which is going to be coated, and therefore a quantitative expression of the adhesion of the film to the support. Measurements of friction coefficients were carried out by Davy[15] and later by Karasev and Izmailova[11] on surfaces of glass and metals following a glow discharge treatment. They found that the friction coefficient of a treated surface is larger than that of the same surface cleaned with a detergent only, or just heated in a vacuum. Thus, the glow discharge offers the best method of 'cleaning' a surface.

This indirect way of estimating the degree of cleanliness of a surface is, however, open to criticism. Indeed, it was found[16] during experiments on adsorption by crystals, that surfaces of germanium and titanium crystals bombarded by argon ions in a low pressure discharge showed high friction values but were not clean. During a brief annealing treatment subsequent to bombardment, a considerable amount of gas was released. This result was interpreted as an indication that argon penetrates the crystal lattice during bombardment, and therefore this bombardment does not cause any degassing.

Quite recently, Kermer[17], has shown that the electron emission of cold metallic surfaces due to an electric field decreased after the surface was subjected to a glow discharge. By this criterion, therefore, the surface has been made 'unclean' by the electric treatment.

The attempt by Bateson[17a] to test the degree of cleanliness of glass by the contact angle between distilled water and the considered surface is not conclusive. He found that glass surfaces exposed to ion treatment and left in the bell jar for 20 min. indicated the same surface conditions as those which were not treated. However, aluminium films deposited onto such surfaces show considerable difference in adhesion.

It is clear from these observations that the notion of cleanliness of a surface has a different meaning for different purposes, and therefore the effectiveness of a cleaning method depends on the process to which it is applied. The friction criterion of cleanliness does not seem to be valid for the deposition process.

Thus, only a direct method, able to indicate the degree of adhesion of a film to its support, would give a truly reliable criterion for the cleanliness of a surface which is going to be coated. It has been suggested by Hass[18] that anodic oxidation provides an excellent means for checking the adhesion of evaporated aluminium films. If the surface onto which the aluminium condenses is not properly prepared, the film will become loose during the anodising process, and blisters will appear. This method, therefore, offers a reliable indication for the cleanliness of a surface to be coated. The difference between aluminium films deposited on treated and untreated glass surfaces may easily be noticed before anodising by the density of pin holes. However, after anodising, this difference becomes more pronounced.

Although the anodising is applicable to aluminium films only, we have employed it in the investigation reported here. A knowledge of the optimum conditions under which this method of cleaning should be carried out would be of great interest, and thus the conclusions arrived at in this particular case may apply generally to metallic and dielectric films.

EXPERIMENTAL PROCEDURE

The glass subtrates—microscopic slides of ordinary shown in this particular case—were given a preliminary treatment and then introduced into the bell-jar of an evaporation plant. There, they were subjected to the action of an a.c. glow discharge in rarefied air under various conditions.

The experimental arrangement consisted of a bell-jar of 45 cm. height and 30 cm. in diameter.

One of the electrodes was an aluminium ring of 21 cm. diameter placed at a distance of 11 cm. below the glass slide under test. Only the downward surface of the glass was taken into consideration. The other electrode consisted of an aluminium plate of 25 cm. diameter placed at a distance of 12 cm. above the glass.

The working pressure was obtained with a conventional oil-vapour pump, giving an ultimate pressure of only 10^{-5}mm.Hg. The evaporation of aluminium was carried out at a pressure of about 10^{-4}mm.Hg, without using a cold trap or drying agent.

As the rate of deposition affects the grain size of the condensed material[19], the time of evaporation for all films was the same, *i.e.* about 18 seconds. This short time was chosen in order to produce high-quality films, since small crystallites are formed only when the evaporation is carried out rapidly and films of small crystallites afford a better means of testing for the required improvement. The films were opaque, having thicknesses between 800 Å and 1,000 Å.

After deposition, the aluminium films were anodically oxidised in a tartaric acid solution of pH = 5.5 for 2 minutes, the operating voltage being 100.

Two procedures were employed for the preliminary cleaning of the glass surfaces:

(1) Washing in chromic acid solution, rinsing in water, washing in warm Teepol solution, and finally rinsing in running tap water.

(2) Rubbing with talc, and then rinsing in running tap water.

It was found experimentally that both procedures give practically the same result, *i.e.* poor adhesion, although Lotmar[20] found an appreciable difference in the structure of films deposited on surfaces cleaned by the two methods. However, the latter procedure is preferable because it can be carried out quickly, and does not attack the glass surface in any way. As we shall see later on, scrupulous cleaning of any kind as a preparatory process to the electrical treatment is superfluous.

The intensity of the electrical treatment was varied, as can be seen below. In order to obtain reliable data on the comparative adhesion properties of films deposited on treated and untreated surfaces, only a portion of every glass slide was exposed to the glow discharge; the remainder was screened by a thoroughly cleaned glass plate.

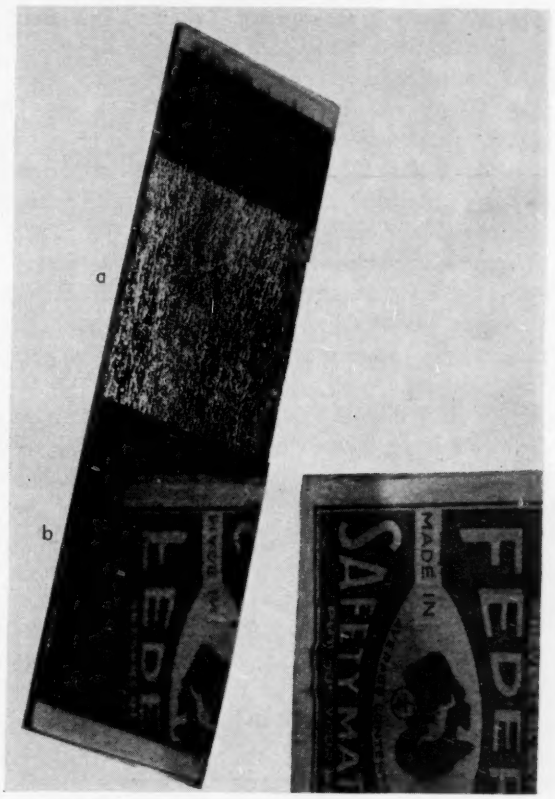

Fig. 1. Appearance of an anodised aluminium film. (*a*) The glass surface was not electrically treated prior deposition; (*b*) the glass surface was subjected to ion bombardment.

EXPERIMENTAL RESULTS

The oxidised films on treated surfaces contained only fine pin holes, the number depending on the film quality, while the untreated portions showed numerous blisters (Fig. 1). These latter films peeled off readily when the substrate surface was badly contaminated.

It follows a detailed description of our experimental conditions and results:

(*a*) A glass slide was subjected to the glow discharge action, then exposed to the atmosphere for 16 hrs. and finally coated with aluminium.

The anodic oxidation of this fim showed that the electric treatment retains its full effect. The effect of the electric discharge remained unchanged also after breathing many times upon a treated surface before coating with aluminium.

(*b*) No preliminary cleaning whatever was given to the glass slides. On the contrary, each slide was effectively contaminated with various organic substances such as Apiezon-C oil, Silicone oil, Apiezon-L grease, butter, glycerine, paraffin, and one was simply rubbed with the fingers. After some hours the excess substance was wiped away with a lintless cloth, and the surface was further rubbed until any visible impurities disappeared.

The film adhesion on such a glass surface after electric treatment was found to be of the same degree as on a surface given preliminary cleaning with talc. However, in the absence of electrical treatment the surface may give a poorer film if not cleaned with talc beforehand.

(*c*) The adhesion of the films was found to be best when the bombardment was carried out at pressures below 0.1 mm.Hg. Films deposited on surfaces subjected to glow discharge at pressures of the order of 0.4–0.5 mm.Hg were of the same quality as films on untreated surfaces.

(*d*) The intensity of the glow discharge is another factor influencing the efficiency of the electrical treatment. The discharge should fill the bell-jar almost uniformly at the level of the glass slide. Then the average discharge density may be calculated from the discharge current and the cross section of the bell-jar at that level.

Three different densities were used: 0.036 mA/cm², 0.09 mA/cm², and 0.13 mA/cm². (The r.m.s. currents and the voltages at the transformer were: 25 mA, 65 mA and 90 mA, and 2,800 V, 3,500 V and 5,000 V respectively). Generally the pressure was kept between 0.035 and 0.050 mm.Hg.

Provided that all other factors were constant, the adhesion of the films increased with increasing discharge density, for bombardment times under 10 minutes.

The increase of the film adhesion with the time of treatment is shown in Fig. 2, where the ordinate represents the ratio

$$\rho = \frac{S_t - S}{S_{10} - S}$$

where S is the net area of an aluminium film, *i.e.* the area of 1 sq. cm. minus the total area of the pin holes and blisters, the glass support being not treated; S_{10} and S_t are similar areas of films deposited onto bombarded surfaces for 10 and t minutes, respectively.

For all three discharge densities mentioned, the optimum adhesions were obtained within 10 minutes. However, considerable differences were found between the effects of various discharge densities for very short periods of the glow action, see Fig. 2.

(*e*) The distance between the ring electrode and the glass slide noticeably affects the quality of the film. This has been shown by placing a glass slide first at 2 cm. and then at 11 cm. from the ring electrode, different areas of the glass being treated in each position. The adhesion of the film was better in the area affected by the discharge when the ring electrode was close to the surface.

(*f*) After the electrical treatment, the affected surface was vigorously rubbed with a perfectly clean cloth. The adhesion of the deposited film was as poor as on the untreated surface. The effect of the electrical treatment, therefore, disappears on rubbing the surface.

(*g*) Electrical treatment carried out for periods of 15 seconds at a time, separated by intervals of 1 to 2 minutes, had the same effect as a treatment over the whole of that time, without interruptions, though in the latter case the glass slide became hotter.

(*h*) A glass plate was placed between the test slide and the ring electrode, so that the latter was not 'seen' by the test glass. The action of the discharge was only slightly reduced, provided that the distance between the two glasses was less than about 10 cm. For distances less than 10 cm., the optimum adhesion was obtained by extending treatment time. When the distance was only 1 cm. the discharge had no effect

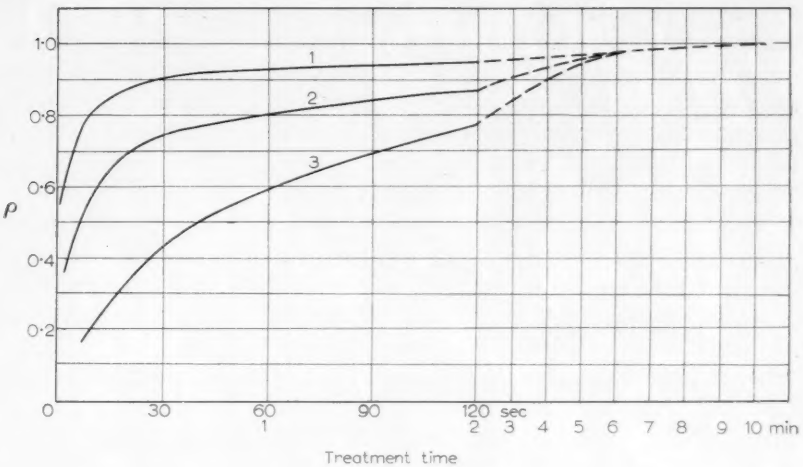

Fig. 2. Increase of the density of condensation nuclei with the time of treatment, for various discharge densities: (1) 0.13 mA/cm.^{2}; (2) 0.09 mA/cm.^{2} (3) 0.036 mA/cm.^{2}.

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on the glass slide, for the same operational periods as those employed in the previous experiment.

The effect of the discharge was slightly reduced when the glass slide was completely screened by an earthed cage of metallic gauze of 1 sq. mm. mesh at a distance of about 2 cm. from the glass.

(*i*) Only treatments of over 5 minutes gave a good stability to oxidised aluminium films.

DISCUSSION OF THE RESULTS

As already described, see Exp. (*b*), glass thoroughly cleaned with detergents, *i.e.* supposedly free of contamination, offers the same adhesion to films after the electrical treatment as surfaces previously contaminated with organic substances, some volatile, others less so. This result does not confirm the suggestion[21] that ion bombardment acts by removing any thin film of a volatile material, evaporation and oxidation accompanying. Indeed, the adhesion was found to be the same with all kinds of contaminating substances, both volatile and non-volatile. This latter conclusion is the only acceptable one. Ennos[22] has demonstrated that in a vacuum system which uses rubber gaskets and grease, and which (as is always the case) has direct access to the oil pump, all surfaces are covered with a layer of adsorbed organic molecules. If one accepts the view that the bombardment removes organic contamination, the cleaning effect would soon disappear after the discharge had been stopped.

The fact that the effect of the treatment persists, in spite of exposing the surface to the atmosphere or breathing on it, see Exp. (*a*), also demonstrates that, for the purposes of coating, a clean surface is not a surface free from any adsorbed layer. It invalidates Ditchburn's[23] assertion that, while the positive ion bombardment cleans the surface, this action is neutralised by a fairly rapid condensation of gas molecules, so that at any one time, a certain fraction of the surface bombarded would be free from gas.*

There is experimental evidence that gases are adsorbed by a surface while being subjected to ion treatment. Oatley[24] has reported that a platinum surface bombarded with positive ions of oxygen traps a considerable amount of this gas in the layers near the surface.

More conclusive is Heavens'[25] finding, that the adhesion of evaporated films on glass plates heated

*No attempt was made to investigate both the effect on visible impurities of prolonged and intense discharges, and the persistence of any change in the substrate surface. The breath figure on a surface as a result of ion treatment may persist for a very long time, as Holland [8a] (page 77) has pointed out, however the effect on film adhesion generally disappears rather quickly.

up to 390°C for periods of up to 44 hours in vacuum, was worse than on glasses which had been subjected to a glow discharge. Therefore, a surface from which the adsorbed gas layers are more or less removed does not offer a better adhesion than a surface bombarded by positive ions.

Previous investigations have emphasised the role of the centres (or nuclei) of condensation, that are present on the target surface prior to the deposition of a condensate, on the formation and the structure of the film obtained[26]. Traub [27] has shown that the presence of such nuclei at a density above a certain value is necessary to obtain films of good quality. Films deposited on surfaces containing a large number of nuclei have fine structures[28], and better adhesion[25]. In these studies the nuclei of condensation were produced by condensing on the substrate atoms of a metal of very low vapour pressure. Recently Wegener[29] has reported that a surface of amorphous quartz, after prolonged heating at 400°C in a high vacuum, had only a limited number of condensation nuclei because of the removal of the adsorbed molecules.

However, such nuclei can readily be created by the ion bombardment of the uncoated surface, as Lotmar[20] has experimentally shown. This preliminary process affects the structure of the film in the same manner as the distribution of the condensing metallic atoms over the surface to be coated. Indeed, Koenig[9] has obtained micrographs showing that lithium fluoride films had very fine structures when deposited onto a carbon substrate previously electrically treated, but a coarse structure if deposited onto a carbon substrate which was not pre-treated in any way. The effect can also be illustrated in a simple manner by reference to the condensation of mercury. The mercury drops are considerably denser and smaller on the treated than on the untreated surface[26].

The above assertion is also supported by the fact that the action of the glow discharge upon the glass surface is achieved instantaneously. Fig. 2 shows that the adhesion of the film is considerably increased if the discharge is only maintained for a second, provided that the current density is not too small. In such a short time none of the actions suggested, such as an appreciable rise in temperature of the substrate, desorption of water, degassing, or decomposition of the grease or oil present, might occur. Thus, by bombarding the surface with positive ions condensation nuclei are created by the charged particles themselves sticking to the surface. These nuclei from which the crystals of the deposited material grow may also be the nuclei of adhesion to the substrate. If these nuclei are removed, by firmly rubbing the surface, see Exp. (*f*), the adhesion of the film is destroyed. The density of the nuclei, which is supposed to be proportional to the adhesion of the film, depends on the number and, possibly, also the speed of the charged particles reaching the surface, as concluded from the Experiments (*d*), (*e*) and (*h*). The fact that a surface screened by a metallic gauze is affected by the glow discharge, see Exp. (*h*), may be explained by the ion diffusion through the meshes.

Experiment (*b*) shows that the presence of a very thin skin of adsorbed molecules of gases, water, grease, etc., does not impede the attachment of ions on the bombarded surface. Since this process takes place independently of the nature of the adsorbed molecules of volatile or non-volatile substances in vacuum, and since these molecules are not affected—evaporated, oxidised, or decomposed—during the normal action of the glow discharge, the object of the patents by Colbert and Weinrich[21], and Morgan[30] is of little practical importance.

The rate of attachment of the ions should be considerable at the beginning of the bombardment. But later, because of the electrical charges already attached to the surface, the sticking of subsequent positive ions becomes more difficult. The curves in Fig. 2 show that the rate of formation of the condensation nuclei is very high at first but drops off as their density increases after the first few minutes. The increase of this density, $d\delta$, in a short bombardment time dt, may then be expressed by

$$d\delta = (\Delta - \delta)\, a\, dt$$

where Δ is the upper limit of the density of the

nuclei, and α is a factor characterising the gas, the target's surface, and the conditions of bombardment. If δ_0 is the density of the nuclei after the initial bombardment, it follows by integrating the above Equ.

$$\frac{\delta}{\Delta} = 1 - (1 - \frac{\delta_0}{\Delta}) e^{-\alpha t}$$

Curves expressed by this Equ. agree with the experimental curves in Fig. 2.

CONCLUSIONS

Objections[31] that by ion treatment the surface becomes rough, and therefore this method should be unsuitable for scientific investigations, are totally unfounded. Observations with the electron microscope at × 30,000 magnification have shown[11] that the treatment by glow discharge does not affect the glass surface in this respect.

The experimental results so far obtained have permitted to arrive at an acceptable explanation of the action of the glow discharge. It remains to extend this investigation and to take into account other factors which control the process.

Usually the treatment is achieved by a d.c. discharge, the cathode being placed in front of the target. An advantage of the d.c. discharge would be the reduction of the time of bombardment. Of interest would be to perform the 'cleaning' process using the bombardment with thermionic electrons. Dunoyer[32] claimed that cleaning by this method is more complete than by ion bombardment.

Investigation of the use of other gases and vapours instead of air, would be of some advantage. If we take the change of the work functions of tungsten and silver surfaces as an indication of the effect of the ion bombardment, then electropositive gases, like Freon and oxygen, would give the best results[33]. It has already been reported that the glow discharge is more effective when the residual gas is air or oxygen[34]. Regarding the use of monatomic gases the results so far are contradictory. While Karasev and Iznisilova[11] recommend argon as the residual gas, Holland[8a] (page 77) has found that aluminium films deposited after treatment in an inert gas "are readily removed from the base."

From numerous practical results, it appears that the method of ion treatment may successfully be applied to any metallic and dielectric films. The position differs with the nature of the target. Friction measurements have already shown that the effect of the ion bombardment is smaller for steel than for glass[11]. We were able to record the fact that the ion bombardment is less effective on an anodised aluminium film than on glass, the process being carried out in the same experimental conditions in both cases.

Regarding the use of various sorts of glasses, no appreciable difference was observed.

The conclusions drawn from these experiments are not less important from the practical point of view. They give the correct procedure of preparing surfaces for deposition of durable and adherent films, at least for aluminium films on glass. The cleaning process as carried out at the present requires various operations which take much time.

To obtain good anodised aluminium films on glass, the following cleaning procedure has been officially recommended: (*1*) Wash glass plates in chromic acid solution for 3–4 hours; (*2*) Rinse in running tap-water, wiping with cotton wool to remove all traces of acid; (*3*) Wash in warm soapy water; (*4*) Rinse in running tap water.

Repeat 2–4 until water flows evenly over the surface. Then: (5) Rub with cotton wool under running tap-water. (6) Dry with linen cloth. (7) Dry thoroughly with another dry linen cloth.

All these operations, however, are quite unnecessary.

In order to effectively remove fine solid bodies which generally cause pinholes, the glass substrate is rubbed with a soft substance non-volatile in the atmosphere, vacuum greases or oils are recommended, and then any visible traces of contamination are wiped away with a lintless cloth. The absence of any breathing pattern indicates that the adsorbed molecules form a uniform coat, and therefore the surface is ready to be subjected to the electric treatment. This bombardment should be carried out

using a discharge density of 0.1–0.15 mA/cm², about 100–150 mA in a 30 cm. bell-jar at pressures below 10^{-1}mm.Hg for 10 minutes.

In conclusion, we may assert that the electric treatment does not 'clean' the surface affected in the usual sense, but it increases considerably the number of condensation nuclei on it.

The experimental part of this investigation was carried out while temporarily engaged in work on Vacuum Physics at C.S.I.R.O. National Standards Laboratory, Division of Physics, Sydney.

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ÜBER HOMOGENE UND HETEROGENE ANTIMON-AUFDAMPFSCHICHTEN

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Summary

ANTIMONY DEPOSITS produced by vacuum evaporation show a variety of structures. Initially, while the deposit is very thin, it is amorphous throughout. Thick films are known to be fully crystallised. Apart from these two homogeneous forms, films of a heterogeneous structure are known. This type of structure was in the past considered to result from a variation of thickness within the film. The author reports on methodical experiments using different evaporation speeds producing deposits of a thickness ranging from 0 to 2,000Å and conducted with the particular objective of studying the crystallisation mechanism in heterogeneous films.

Sommaire

LES DEPOTS d'Antimoine évaporés sous vide ont différentes structures. Tout d'abord, le dépôt est mince et amorphe. Les depôts épais sont connus comme étant complêtement crystalisés. A part ces deux formes homogènes, il existe des dépôts de structure hétérogène. Ce type de structure était considéré comme dû à des variations d'épaisseur du dépôt. L'auteur décrit les essais méthodiques faits à différentes vitesses d'évaporation, produisant des dépôts d'épaisseur comprise entre 0 et 2000Å, ceci dans le but d'étudier le mecanisme de la crystalisation dans les dépôts hétérogènes.

EINLEITUNG UND PROBLEMSTELLUNG

WIRD ANTIMON IM Hochvakuum aufgedampft, so entstehen je nach Versuchsführung Schichten mit verschiedenen Eigenschaften. Es kondensiert zunächst in nichtmetallischer Form; bei Zimmertemperatur ist jedoch nur die kristallisierte Modifikation stabil[1-4]. Wie schnell der Übergang in die metallische Modifikation stattfindet, hängt von den Versuchsbedingungen ab. Die aufgedampften Schichten können also einheitlich aus nichtmetallischem oder metallischem Antimon aufgebaut sein ('homogene Schichten') oder es können beide Antimon-Modifikationen nebeneinander vorhanden sein ('heterogene Schichten'). Ob eine homogene oder heterogene Schicht vorliegt, kann unschwer festgestellt werden, da im durchfallenden Licht die homogene Schicht bei Vorliegen der nichtmetallischen Modifikation gleichmässig braun, bei Vorliegen der metallischen Modifikation gleichmässig blau erscheint. Haltbare heterogene Schichten zeigen braune Flecken in blauem Umfeld.

Der Umwandlungsprozess der nichtmetallischen Antimon-modifikation in die metallische wurde bereits von verschiedenen Forschern beschrieben[5, 6]. Heterogene Schichten untersuchten insbesondere Lotmar[7] und Götzberger[8]. Die Heterogenität wird von diesen Autoren auf Unterschiede in der Schichtdicke zurückgeführt. Eigene Photometermessungen[4] anlässlich einer früheren Arbeit führten zu der Annahme, dass die Schichten keine Dickenunterschiede aufweisen, sondern dass das Auftreten von braunen Flecken auf blauem Umfeld durch kristallisations-hemmende Oxyd- bzw. Adsorptionsschichten zwischen einer bereits kristallisierten und einer nachträglich aufgedampften nichtmetallischen Schicht zustande kommt.

Die Stabilität der nichtmetallischen Phase wird begünstigt durch amorphe Unterlagen, Fremdstoffeinbau in der Schicht[9], tiefe Trägertemperatur und

* MS. received December, 1957.

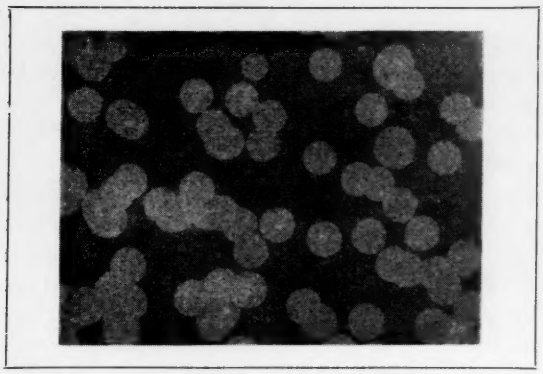

Abb. 1. In Umwandlung befindliche Antimonschicht. Helle Kreise: Kristallisierte Phase. Dunkles Umfeld: Amorphe Phase.

Abb. 2. Heterogene Antimonschicht nach Beendigung der Kristallisation. Eine Stunde bei 300°C nachträglich getempert.

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geringe Schichtdicke. Der Übergang in die metallische Form wird erleichtert durch kristallisierte Unterlagen, möglichst grosse Reinheit der Schicht, höhere Temperatur und grössere Schichtdicke. Die Mindestschichtdicke, bei der die Umwandlung einsetzt, ändert sich ebenfalls mit den Versuchsbedingungen (Temperatur, Reinheitsgrad und Aufdampfgeschwindigkeit). Die Umwandlungsgeschwindigkeit nimmt mit steigender Schichtdicke zu und verläuft bei Schichtdicken von über 2,000Å explosionsartig[8, 6].

VERSUCHSERGEBNISSE BEI SCHICHTEN VERSCHIEDENER DICKE

Im Nachfolgenden wird eine Übersicht gegeben

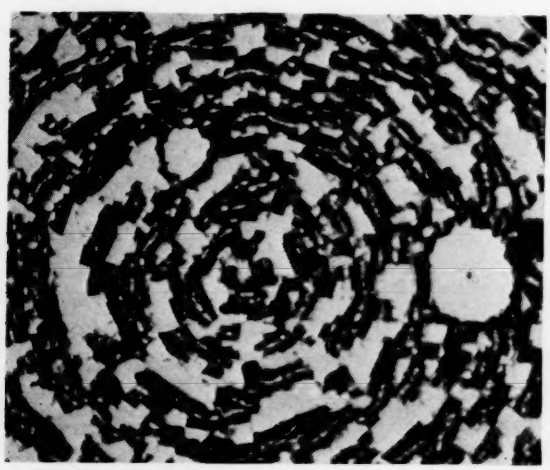

Abb. 3. Explosionsartig kristallisierte Antimonschicht. Weiss: Auf der Unterlage haftende Kristalle. Dunkel: Von der Unterlage abgehobene Schichtstreifen. Schichtdicke 2,200 Å. Auflichtaufnahme.

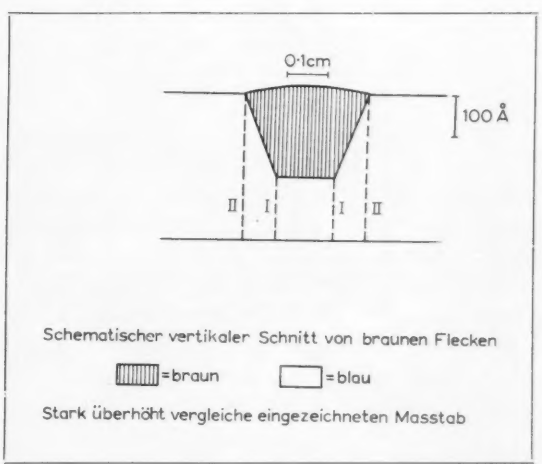

Abb. 4. Schematischer vertikaler Schnitt von braunen Flecken (In vertikaler und horitzontaler Richtung verschiedener Massstab).

über die Veränderung in der Schichtstruktur während und nach dem Aufdampfprozess. Wegen des grossen Einflusses, den die Aufdampfgeschwindigkeit auf die Eigenschaften der entstehenden Schicht ausübt, werden im folgenden die bei hohen Aufdampfgeschwindigkeiten ($v > 100$ Å/sec.) und die bei niedrigen Aufdampfgeschwindigkeiten ($v \leqq 1$ —100 Å/sec.) erhaltenen Ergebnisse getrennt behandelt.

Folgende Versuchsbedingungen wurden in allen Versuchen konstant gehalten: Grösse und Material des Trägers: Glasplatte von 25 cm², die Trägertemperatur 20°C und das Aufdampfvakuum $\sim 10^{-5}$ mm. Hg.

Schnell Aufgedampfte Schichten

($v > 100$ Å/sec.)

(*a*) *bis 140 Å*: Amorph (braun), elektrischer Widerstand/cm.² $> 10^{10}\,\Omega$, auf der Unterlage gut haftend.

(*b*) *von 140 bis 700 Å*: Anfänglich amorph (braun), kurz nach Abbruch der Bedampfung Auftreten von mehreren Kristallisationszentren, von denen aus die Kristallisation mit konstanter Geschwindigkeit fortschreitet. Es ergeben sich dadurch blaue Kreise auf braunem Grund (Abb. 1). Geschwindigkeit des Fortschreitens der blauen Grenze: 0.5 mm./min. bei 140Å, 5 mm./min. bei 400 Å. Endzustand: Homogen blau gefärbte Schicht, gut haftend auf der Unterlage. Elektrischer Widerstand/cm.² bei 140 Å $\simeq 10^4\,\Omega$, bei 500 Å $\simeq 100\,\Omega$.

(*c*) *von 700 bis 2,000 Å*: Anfänglich amorph (braun), wandelt sich wie unter (*b*) angegeben um mit einer Geschwindigkeit von ~ 1cm./min. Endzustand: Nicht mehr transparent, sehr gut haftend, elektrischer Widerstand/cm.² $\simeq 10\,\Omega$.

(*d*)*über 2,000Å*: Herstellbar nur bei hoher Aufdampfdichte, d.h. bei kleinem Abstand zwischen Verdampfungsquelle und Träger. Plötzliches Auftreten eines Umwandlungszentrums, von dem aus die Umwandlung explosionsartig fortschreitet[8, 6].

Langsam Aufgedampfte Schichten

($v < 1$—100 Å)

(*a*) *bis 180 Å*; gleiche Eigenschaften wie unter (*a*) oben.

(*b*) *180 bis 700 Å*: Anfanglich amorph (braun). Die unter (*b*) oben beschriebene Kristallisation beginnt hier schon wahrend der Aufdampfung. Die Kristallisationskreise erscheinen hier jedoch nicht blau, sondern gelblich und heben sich nur mehr schwach vom braunen Umfeld ab. Bei Abbruch der Bedampfung schreitet die Kristallisation wie bei (*b*) im vorigen abschnitt weiter fort, wobei die dadurch gebildete Schicht blau erscheint. Endsuztant: Braune Kreise in blauem Umfeld (Abb. 2). Das blaue Umfeld hat die gleichen Eigenschaften wie unter dem obigen (*b*) angegben. Die braunen Fleckenerswisen sich als heterogen zusammengesetzt, zeigen in weissem Licht grossere Transparenz, ausserdem grosseren elektrischen Widerstand und bessere Haftfestigkeit als das blaue Umfeld (vgl. vorhergechenden Abschnitt). Die Flecken werden umso grosser, je langer die Aufcampfung nach dem Auftreten der Ksirtallisationszentren fortgesetzt wird.

(*c*) *über 700 Å*: Diese Schichten sind nicht mehr transparent. Sie besitzen nach dem Umwandlungsproess metallischen Glanz und gute Haftfestigkeit. Die unter (*b*) erwähnten braunen Flecken machen sich durch eine geringere Reflexion an der Oberfläche der Schicht bemerkbar.

(*d*) *über 2,000 Å*: Als homogene amorphe Schicht durch langsames Aufdampfen nicht darstellbar, da vorher der Endzustand von (*b*) erreicht wird. Man kann aber die für die Explosion notwendige kritische Schichtdicke des amorphen Antimons durch zunächst langsames, später rasches Aufdampfen erhalten. Es ergibt sich dann das in Abb. 3 dargestellte Bild: Überlagerung der langsamen Kristallisation nach (*b*) (weisse Kreise) und einer entsprechend (*d*) auftretenden Explosion, wobei die Schicht teilweise abblättert (kreisförmig angeordnete, in der Aufnahme je nach Orientierung hell oder dunkel erscheinende Kristalle).

Tabelle 1 bringt eine Übersicht über die Antimonschichten verschiedener Dicke und Herstellungsart.

Spezielle Untersuchungen der Heterogenen Schichten

Wie unter (*b*) in beiden Fällen oben beschrieben,

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Tabelle I. Übersicht über die Eigenschaften von Antimonschichten verschiedener Dicke

Schichtdicke Å	*Bedampfung*	*Bedampfungszeit* sec.	*Modifikation*		*Umwandlungsgeschwindigkeit*	*Farbe im Durchlicht*		*Elektr. Widerstand* Ω/cm.²	*Haftfestigkeit auf Glas*
			Anfangszustand	*Endzustand*		*Anfangszustand*	*Endzustand*		
bis 180 (±20)	langsam	300–400	amorph	amorph	—	braun	braun	$<10^6$	gut
180–700	Abbruch sofort nach Beginn der Umwandlung	300–400	amorph	metallisch	zunehmend mit der Schichtdicke: 0.5 mm./min. bei 180 Å, 5 mm./min.[3] bei 400 Å	braun	blau	$\sim10^3$	gut
	Abbruch 5–10 min nach Beginn der Umwandlung	600–1,000	amorph	amorph und metallisch		zunächst braun, dann: blaue Flecken im braunen Umfeld	braune Flecken in blaume Umfeld	$\sim10^2$	gut (braune Flecken weniger gut als blaues Umfeld)
700–2,000	Beschleunigt nach Beginn der Umwandlung	bis 180 Å wie oben, dann: 1–5	amorph	metallisch	~1 cm./min.	nicht transparent		~10	gut
über 2,000	sehr schnell	1–5	amorph	metallisch	explosionsartig			—	Schichten reissen und blättern ab

Tabelle II. Schichtdicken (in Å) von Heterogenen Antimonschichten (im Hochvakuum auf Glas aufgedampft[6]

Gemessen nach Tolansky-Methode[4,10]		*Berechnet aus Titrierter Menge*
Im Zentrum der Braunen Flecken	*Mittelwert der übrigen Schicht*	
287	280	281
303	294	291
358	353	359
409	391	397
610	602	598

Tabelle III. Widerstandsmessungen an Heterogenen Antimonschcihten[6]

Schichtdicke nach Tolansky Å	*Widerstand in Ohm, Prüfstrecke 2 mm.*		*Aus dem Widerstand berechnete Schichtdicke der Blauen Schicht unter dem Braunen Flecken.* Å
	im Blauen Umfeld	*im Braunen Flecken*	
280	60	90	187
~280	55	95	163
375	30	65	173

lassen sich Antimonschichten erhalten, bei denen braune Flecken auf blauem Grynde afutreten (Abb. 2). Untersucht man diese Flecken auf ihre Transparenz in demjenigen Wellenbereich, in dem die Transparenz der beiden Modifikationen gleich ist (414 mu), so ergibt sich kein Unterschied in her Lichtdurchlassigkeit und somit kein Unterschied in der Schichtdicke[4, 6]. Die Schichtdicke konnte ferner mit der Tolansky'-schen Vielfach-Interferenzmethode[9, 10] in den Flecken und im Umfeld sehr genau bestimmt werden[6].

In der Tabelle 2 sind die Messergebnisse dargestellt. Es ergibt sich lediglich eine geringe Vergrösserung der Schicht im Zentrum des braunen Fleckens, die aus dem Unterschied der Dichten der beiden Modifikationen leicht erklärbar ist. In der letzten Spalte der Tabelle sind Werte aufgeführt, die durch chemische Bestimmung der aufgedampften Antimonmenge (bromatometrische Titration) ermittelt wurden. Beide Werte zeigen eine ausgezeichnete Übereinstimmung. Tabelle 3 zeigt Mikromessungen des elektrischen Widerstandes im braunen Flecken und im blauen Umfeld.

Die Untersuchungen ergeben für die Struktur der heterogenen Antimonschicht das folgende Bild (vgl. Abb. 4): Unter dem braunen Flecken befindet sich eine 150-200 Å dicke homogene kristallisierte Antimon-Unterlage. Die gestrichelte Linie I in Abb.5 zeigt die Kristallisationsfront zu Beginn der Fleckenbildung an. Von dieser Grenze schreitet die Kristallisation nun in horizontaler Richtung weiter fort, während die auf der Schicht sich niederschlagende Antimonmenge in vertikaler Richtung amorph aufwächst. Es entsteht somit die in Abb. 4 gezeigte Form der Einlagerung von amorpher Phase in die kristalline Phase. Die gestrichelte Linie II zeigt die Kristallisationsfront bei Beendigung des Aufdampfvorganges an. Von dieser Front aus schreitet nun die Kristallisation durch die gesamte Schicht weiter fort bis zur völligen Umwandlung des ausserhalb des gebildeten Fleckens befindlichen Antimons. Dass sich die Kristallisation nicht über die (ausgezogene) Grenzschicht zwischen amorpher und kristalliner Phase ausbreitet, lässt sich—wie bereits eingangs erwähnt—durch eine Oxyd- bzw. Adsorptionsschicht erklären, die sich beim Kristallisationsvorgang auf der Grenze zwischen Kristall und Gasraum ausbildet.

Der Gerätebauanstalt Balzers, Liechtenstein, danke ich für die Gewährung eines Forschungsstipendiums und für die Bereitstellung der Aufdampfapparatur.

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THE PREPARATION OF ELECTROLUMINESCENT PANELS

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Summary

ALTHOUGH MUCH has been written on the theory of electroluminescence there is little practical data available which would assist in the construction of a panel. This report gives complete details of the manufacture of electroluminescent light sources, using vacuum deposited films for the conducting electrodes. Commencing with a glass base, which is made electrically conductive by sputtering a transparent cadmium oxide film, the phosphor is mixed with a resin based lacquer and sprayed on the semiconducting oxide film. This is followed by a second layer of insulating material upon which the top electrode of aluminium may be deposited by evaporation in vacuum. Upon applying an alternating field between the two conducting electrodes, the phosphor emits light through the transparent oxide film. Three power supplies have been made to supply alternating current at suitable voltage and frequency, and full details are given.

Sommaire

BIEN QUE L'ON AIT beaucoup écrit sur la théorie de l'électroluminescence, il y a peu de données sur la réalisation pratique de panneaux luminescents. Cet article donne des détails complets sur la fabrication de sources lumineuses, employant la méthode d'évaporation sous vide pour les électrodes de contact. Le panneau de verre est rendu conducteur par un film transparent d'oxyde de cadmium déposé par projection cathodique; du phospore est mélangé à une laque à base de résine et projété sur le film d'oxyde semiconducteur. Ensuite, une couche isolante est déposée et aux deux extrémités on dépose sous vide les deux électrodes d'aluminium. Lorsque l'on branche un champ alternatif entre les deux électrodes, le phosphore émet de la lumière au travers du film transparent d'oxyde. Trois alimentations de courant alternatif délivrant la tension requise à la fréquence necessaire ont été construites et sont décrites.

INTRODUCTION

DURING THE PAST few years considerable interest has been aroused in electroluminescence, which was first observed by Lossev[1] as a flash of light emitted upon applying a d.c. field to a crystal of silicon carbide. Light was emitted only whilst the field was changing, and not under steady conditions. Electroluminescence can be defined therefore as the production of light by direct excitation of a phosphor under the influence of a changing electric field. Gudden and Pohl[2] as early as 1920 had observed that an electric field could influence the light emission from a phosphorescent zinc sulphide, and after the discoveries of Lossev, it was this type of phosphor which later workers found to be most useful. Destriau[3] in 1936 used an alternating potential in order to obtain a continuous emission of light, and he found that light could be produced although there was no direct electrical connection to the phosphor. He suspended fine particles of the phosphor in oil and resin films, and by applying an alternating field he was able to make the first practical light source.

Many different phosphors have been investigated[4] but few have given a useful light output at normal mains voltage and frequency. However, specially prepared zinc and cadmium compounds have proved fairly effective. The compound most commonly used consists of zinc sulphide containing small quantities of such metals as copper, aluminium, manganese and lead. These are known as activators, and they produce the impurity centres in the structure of the phosphor upon which the emission of light is dependent[5].

* MS received June 1958.

Phosphors which emit light under the influence of ultra-violet or x-radiation have also been produced. These are known as photo-electroluminescent phosphors, and they are excited by short-wavelength radiation falling on layers which have a d.c. field across them. A photo-electroluminescent phosphor has been used to make an image converter for intensifying ultra-violet or x-ray patterns. This has proved useful in x-ray fluoroscopy, where it is necessary to keep the x-ray level low to avoid injury to a patient.

The uses of electroluminescence have now been extended, and no longer is it considered to be merely a novel light source. An electroluminescent panel has been used in combination with a photo-conductive layer as a light amplifier to increase the brightness of an image by several times [6, 7]. A variety of different combinations of photo-conductive and electroluminescent layers have been produced by the Sylvania Company for use in electro-optical switching systems and for storing the information from digital computers. It has been suggested that electroluminescent panels should also have important application to the general problem of displaying information, for example in radar and air traffic control. Although much has been written on the theory of electroluminescence, and its applications, little has been published on methods of production. The patent literature is extensive but is generally so wide in scope as to be of little value to the would-be manufacturer. This report has been written to assist those who need to make electroluminescent panels, and who have available vacuum deposition plant. Electroluminescent phosphors can be obtained commercially†, and the panels described here have been made using vacuum deposited conducting films for the electrodes. With the recent advances in reactive sputtering techniques it is possible to make conducting transparent films on glass, which in conjunction with evaporated aluminium layers, can be used for the conducting electrodes of the lamp. However, lamps based on chemically formed conducting transparent glass are already in production and are likely to be more economic for use in large-scale manufacture.

† Thorn Electrical Industries Ltd.

PANEL CONSTRUCTION

A Typical Lamp

The practical electroluminescent lamp is effectively a parallel plate capacitor with the phosphor suspended in the dielectric between conducting electrodes. A lamp may be constructed from a sheet of glass which has the following layers on one side:

(i) A conducting transparent film in contact with the glass.
(ii) A layer of phosphor powder suspended in a dielectric.
(iii) A conducting back electrode.

Panels have already been produced using a transparent conducting glass which is commercially available. This is made by spraying glass, heated to a temperature near its melting point, with a solution of a suitable metallic salt such as stannous chloride[8]. The conducting coating is between 0.1 and 0.5 microns thick and may have a resistance of 80-100 ohms per square with an optical transmission of 85%. The disadvantage of this material is firstly that it is not uniform in conductivity over the surface and secondly it is very difficult to remove. The removal of the film is sometimes necessary for etching a pattern in the conducting layer or for making a contact spot for the top electrode. The baking at high temperature can also set up undesirable strains in the glass.

The layer of phosphor has been applied in several ways, either by settling from a suspension of the powder, or by spraying the powder mixed with a resin based lacquer to make a paint. The solvents are then baked out of the lacquer leaving the phosphor layer dispersed throughout a dielectric resin film. Layers of uniform thickness may be obtained by this method. In the case of the settled layer of phosphor, the dielectric resin is sprayed separately.

The ideal properties of the dielectric are:

(i) High dielectric constant.
(ii) High breakdown strength and good insulation resistance.
(iii) No reaction with the phosphor.
(iv) Good physical and chemical stability.

The properties of some epoxy-resins conform

closely to the above and melamine formaldehyde resins have also been used with success. Some silicone resins are excellent but are very expensive. The dielectric constant of all these is rather on the low side, and the performance of the lamp has been greatly improved by loading the lacquer with finely powdered material of high dielectric constant such as titanium dioxide or barium titanate[9].

The back electrode of the lamp has been made by pressing on a thin metal foil or by spraying a conducting paint made from colloidal graphite or metal powder. Neither of these methods are completely satisfactory because when electrical breakdown occurs the coatings are burnt away to form a black spot, and the plate may be permanently short-circuited.

It has been found generally advisable to protect the completed lamp by spraying on a thick water-repellant coating on the back surface. Water vapour has a slow adverse effect on the phosphor and resins, causing the light output to gradually decrease with age.

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A Lamp with Vacuum Deposited Electrodes

Two types of vacuum deposited conducting films were tested as the transparent electrode in an electroluminescent panel. These were:—

(i) thin gold films deposited on bismuth oxide base layers, and

(ii) semiconducting cadmium oxide films prepared by reactive sputtering.

It was discovered that although the gold films had a much higher conductivity for a given optical transmission, they were not always capable of carrying the current which was demanded as the reactance of the plate decreased at higher frequencies. Breakdown of the film occurred particularly after several minutes operation when the lamp reached its normal running temperature. The cadmium oxide films were at least ten times thicker, being of the order 1,200 Å thick. No instances of breakdown were observed in cadmium oxide films used at current densities up to 10^4 A/sq. cm. of cross-sectional area.

Opaque evaporated aluminium films were found to be satisfactory for the back electrode. They were capable of carrying the current but burnt away ('self-healed') if breakdown in the dielectric occurred, leaving a small pinhole just visible under close observation.

The construction of an improved panel is shown in Fig. 1. Between the phosphor layer and the aluminium back electrode is a thin dielectric film of synthetic resin. The use of the separate dielectric layer is advantageous in preventing electrical breakdown, and has been found necessary to give a useful life to a lamp running at a high field strength for maximum light output. It is thought that the particles of phosphor may be responsible for breakdown in lamps not having a separate insulating layer, because the dielectric strength of the phosphor may not be as high as that of the resin in which it is embedded.

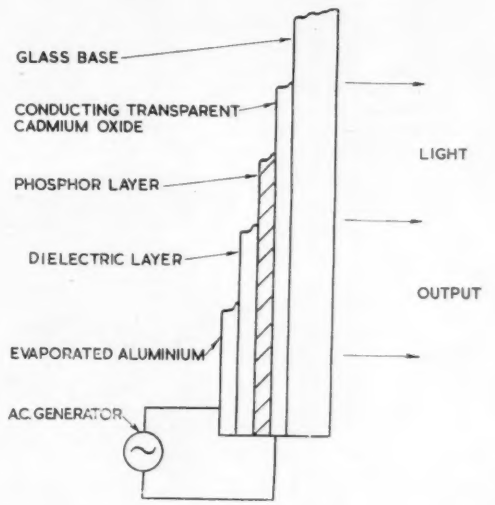

Fig. 1. Diagram showing the construction of an improved type of electroluminescent panel.

PANEL MANUFACTURE

A description of the manufacture of an electroluminescent panel in our laboratories is given below in five processing steps.

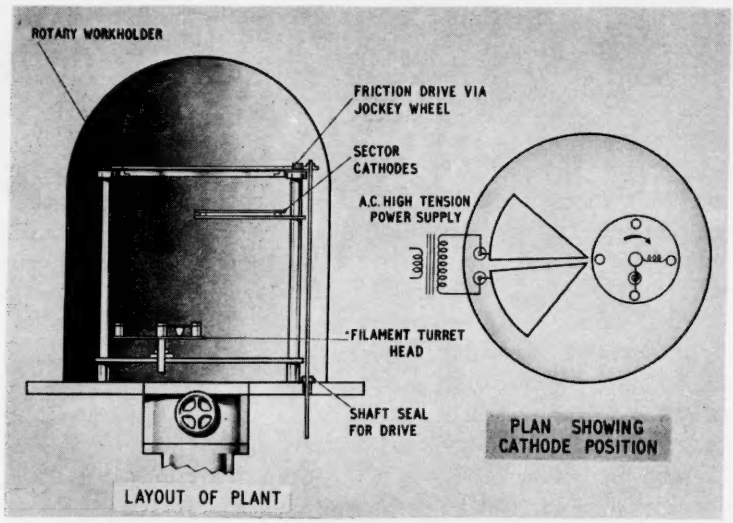

Fig. 2. Diagram showing the layout of the cathode sputtering and evaporation plant. The apparatus consists of one or more pairs of sector cathodes mounted under a plane rotary work holder. Each pair of electrodes can be connected in turn to an a.c. high voltage supply. Each sector functions alternately as an emitting cathode during sputtering. The turret head of vapour sources enables multilayer coatings of evaporated and sputtered films to be made in one vacuum cycle.

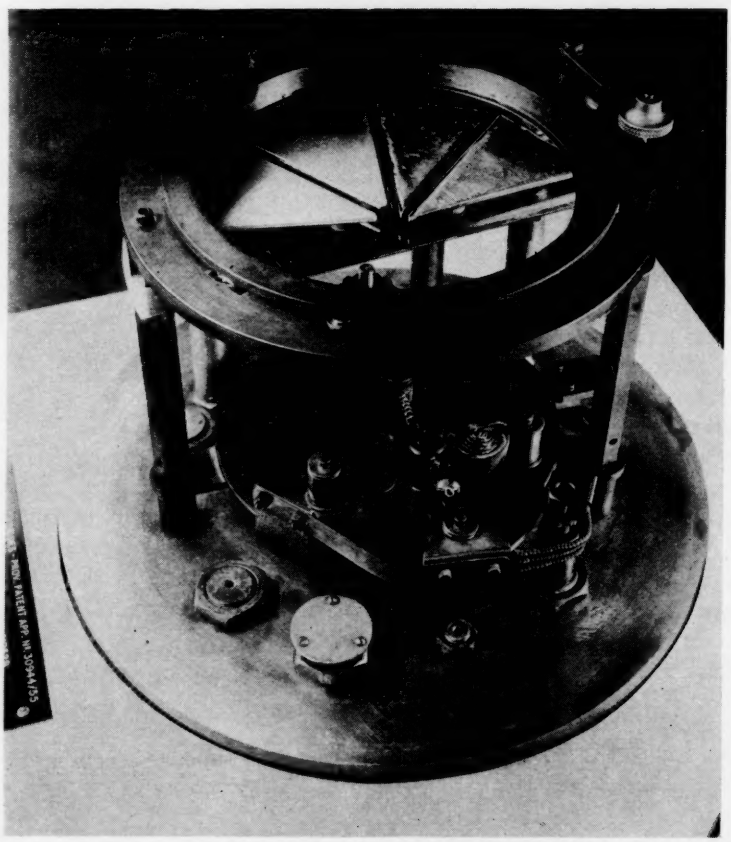

Fig. 3. Photograph of the plant used for the vacuum deposition of conducting films.

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The Conducting Transparent Film

The transparent-conducting film is prepared by sputtering cadmium oxide to produce a film with a surface resistance less than about 1,000 ohms per square and with a transmittance of about 75%. If sputtering is continued until the cadmium oxide film is coloured magenta by reflection, then the optical transmission for green light will be at its theoretical maximum of about 85%. This corresponds to an optical thickness of a half-wave length for green light. Assuming the refractive index to be about 2.0, the physical thickness is of the order 1,200 Å. Under these conditions, the resistance can be as low as 75 ohms per square. For small panels, the resistance value is not important, provided it is less than about 1,000 ohms per square but for very large panels it is advisable to make the film resistance as low as possible on account of the increased current flowing.

The method of reactive sputtering has been completely described elsewhere[12-14] but sufficient detail will be given here to enable a film to be sputtered.

A suitable sheet of glass is thoroughly washed with a detergent and water, and then vapour-degreased in iso-propyl alcohol. It is then placed on an uncooled support in the sputtering work chamber, parallel to the water-cooled cadmium cathode and spaced from it by a distance corresponding to about one-tenth of the cathode diameter. The system is then pumped to a pressure better than 1 micron Hg on the diffusion pump, and when this pressure has been obtained, the pump is baffled down and a gas mixture which has been prepared previously is admitted to the chamber *via* a fine-control needle valve. The h.t. is switched on, and gas is admitted until a glow discharge appears. Then it is maintained at a rate sufficient to hold the pressure at a value corresponding to the best conditions for sputtering. The sputtering pressure usually lies between 10 and 100 microns of Hg, but the controlling factor is the cathode dark space which must extend from the cathode to approximately three-quarters of the distance from cathode to work. If the dark space is much less, then the sputtering rate will be low, since metal is returned to the cathode by collision with gas molecules. On the other hand, when the dark space encloses the work, the work surface may become overheated by intense electron bombardment, and also the discharge may not be uniformly maintained over the cathode surface. Using a d.c. sputtering system as described above, a potential of 3 KV is required.

A suitable film of cadmium oxide, already in a high state of conductivity can be prepared by sputtering for about 7 minutes at a current density of 1mA/sq. cm of cathode surface, in a gas mixture of 5% oxygen, 95% argon. By using this mixture, the film is deficient in oxygen, thus providing free cadmium atoms in the crystal lattice, upon which the semiconducting properties of cadmium oxide are dependent. If it is not convenient to use a gas mixture, the film may be sputtered in pure oxygen. An almost non-conducting completely oxidized film will be produced which can be made electrically conducting by heating to 450°C in air, when the oxide dissociates to give an excess of cadmium atoms in the lattice.

Certain advantages can be gained by using an improved method of sputtering[15] requiring alternating current. The apparatus is shown diagramatically in Fig. 2. In this method a pair of water-cooled cadmium cathodes are used, each one shaped as a sector of a circle. They are connected directly to the output of a h.t. transformer, and the work is rotated parallel to and concentric with them. During each half-cycle one sector will be negative with respect to the other; it will therefore be bombarded with positive ions and will emit sputtered atoms which are uniformly deposited on the work. In all other respects, the operation of sputtering in an a.c. discharge is similar; the dark space is visible from each sector and the discharge is controlled in the same way.

The sputtering rate is approximately equivalent if one uses a current density based on the total circular area of the work and not on the area of the sector cathodes themselves. The angle of the sectors is not critical, but for maximum uniformity the sectors should be placed diametrically opposite and have as large an angle as possible in order to obtain normal emission from the points. The sectors can also be operated next to each other; this is often the most convenient arrangement, but there is a slightly increased sputtering rate from the point of the sector

under these conditions. The advantages of this system of sputtering are that no rectifiers are required, and that the whole of the work surface is not covered by cathode. This enables vapour sources to be used for evaporation in sequence with sputtered layers. Several pairs of sectors may be used for the sputtering of different layers in the same vacuum cycle, or one pair of sectors may be composed of different materials for the sputtering of mixtures or alloys with a preferred composition. The system is therefore very convenient for the production of multilayers of sputtered and evaporated films. The chamber details of a typical 12 inch. plant are shown in Fig. 3. This plant was used for carrying out the vacuum deposition described in this report.

Cadmium oxide films will deteriorate if left exposed to the atmosphere. It is therefore best to spray the phosphor layer within a few days after the conducting surface has been deposited. Should there be any evidence of reaction between the cadmium oxide and a particular phosphor, then a very thin lacquer film can be sprayed on before the phosphor.

Conducting contact strips can be made along the edges of the glass plate by the evaporation of a thick film of aluminium, by spraying a fusible alloy of bismuth/tin, or by a soldering technique using pure indium. Contact strips of chemically deposited silver can also be baked on before work is commenced on the plate. A combination of these methods is often advantageous.

The Phosphor Layer

In order to cover one square foot of glass with a phosphor layer, the following procedure was carried out. 8 g. of a suitable electroluminescent phosphor*, finely milled and dried, was mixed to form a smooth paint with 10 c.c. of a melamine modified alkyed resin-based lacquer† and 5 c.c. of thinners. This was sprayed by hand using an air pressure gun at 40 p.s.i., with the electrodes masked using a strip of adhesive tape. This tape was removed before baking out the plate at 140°C for 30 mins. to cure the resin.

The Dielectric Layer

Two possibilities exist for this. The dielectric layer may consist of melamine resin as above, in which case a similar quantity of lacquer and thinners is sprayed and cured as a separate layer without the phosphor. Alternatively, a 'Melinex' sheet, for example 0.0005 inch thick, may be laminated to the uncured phosphor and resin layer. This is done by baking out the solvents at 60°C for 10 minutes, then laminating the foil to the sticky resin layer using a rubber roller. The composite layer is cured at 140°C for 30 mins. as before.

The light output of the lamp is increased by loading the resin layer with finely powdered material of high dielectric constant. This increases the electric field across the phosphor layer. A suitable mixture for spraying is obtained by mixing 6 g. of titanium dioxide powder with 10 c.c. of lacquer and 5 c.c. of thinners. This is sprayed and cured in the same way.

The Aluminium Evaporation

An opaque film of aluminium should be evaporated through a suitable mask (to give a desired pattern) at a pressure better than 0.5 micron Hg. The evaporation can be carried out on the 'Melinex' foil after lamination, or upon an otherwise completed panel. In the apparatus shown in Fig. 3, 3 inches of 20 s.w.g. pure aluminium wire were evaporated to deposit an opaque highly conducting film. Contact can be made to the aluminium film either by pressing a metal foil lightly against the back of the panel, or by arranging for a special contact strip of sprayed metal along one edge of the plate not in electrical connection with the original conducting transparent layer.

Protective Backing

In experimental panels, adhesive tape has been used to provide a protective backing, but this is not permanent enough. The panel prepared complete with contact leads, should be sprayed over the back and edges with a matt bituminous protective paint, to keep out all moisture which has a very slow deleterious effect on the dielectric properties of the resins.

PANEL POWER REQUIREMENTS

Although maximum light output for a given phos-

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phor may be obtained at an input frequency of about 10 Kc/s., the electrical efficiency is low under these conditions. The power factor is very low, and as the frequency increases, the reactance may reach a value of a few hundred ohms for a panel of capacitance 0.1 μF. A panel size 10 $\times$ 8 inch. has a capacitance of the order 0.03 μF, so that an increase in size up to 2 $\times$ 1 ft. would give a capacitance of 0.1 μF with a reactance of the order of 300 ohms at 5 Kc/s. This involves the construction of a power supply with a low impedance output, giving nearly 1A at 270V. At frequencies of the order 500 c/s. to 1 Kc/s., the light output is still fairly good and the efficiency is higher. Efficiencies of 9 lumens/watt have been obtained, compared with 16 lumens/watt for a tungsten filament lamp. Frequencies of this order are thus most convenient to use.

A field strength of at least 5×10^4 V/cm. is required across a dielectric which is just over 0.002 inch total thickness. This means that the panel must run at 250-300 V (r.m.s.), and the dielectric is under considerable electrical stress. Nevertheless, both the melamine resins and the 'Melinex' polyester foils have proved to be reliable.

It can be shown that because of the small but finite time of persistence of the phosphor, (5-10 μ sec.) a steep-sided pulse with a flat top is more efficient than a sinusoidal pulse from the point of view of light output. Light continues to be emitted when the field is no longer changing, thus, by using a flat-topped pulse there is no waste of energy during the persistence time of the phosphor.

The above fact is very convenient in design, since a suitable square-wave oscillator is simple to make and is efficient in operation. Commencing with the initial requirement of 250-300 V r.m.s. at 1 Kc/s., three power supplies were designed and made. A valve oscillator with an output of nearly 40 watts was designed for general experimental work, and two transistor oscillators, operated from pocket torch batteries, were made for demonstration purposes. These had outputs of 2.7 watts and 125 milliwatts respectively. The smaller power supply was about the size of a match box and was capable of giving enough current to operate a small electroluminescent sign which could be carried in the lapel. Full details of these power supplies are given below.

40-Watt Oscillator

This is a ringing-choke oscillator using two beam tetrodes type 807 in parallel. The circuit diagram is given in Fig. 4. Compensation for the different valve characteristics is provided by R_1, and the output voltage may be varied from about 100 V to 250 V (depending on the load) by adjustment of the cathode

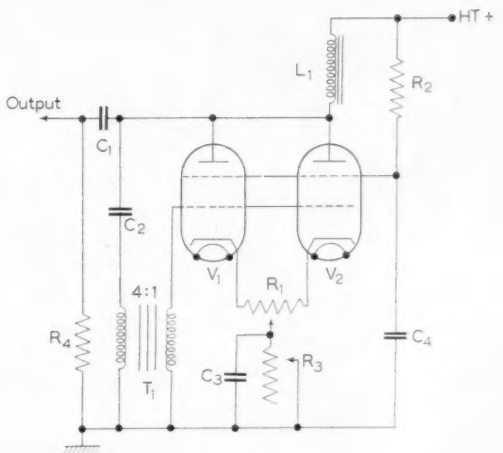

Fig. 4. Circuit diagram of a 40-watt valve oscillator.

bias resistor R_2. Feedback is obtained by inductively coupling the anode and grid circuits using a standard intervalve coupling transformer with a turns ratio of 4 : 1.

Frequency is mainly dependent on the constants of the anode circuit, but the loading of the oscillator and the setting of the bias control also have some effect. When the oscillator is operating at no-load, very high voltages of at least 1 kV are produced, and this may be harmful to the circuit components. The oscillator should not be used without a load unless allowance for this effect has been made in design. The voltage regulation of the output is not good, but the primary requirement was for a cheap and efficient oscillator with a fairly low impedance output. The output of the oscillator is about 160mA at 250V and about 120mA

at 300V (r.m.s. values). The waveform is steep-sided with an almost flat top. When using the components specified, the output frequency lies between about 1 Kc/s. and 2 K/cs. according to the setting of the bias control and the capacitance of the load, which should not be greater than 0.1 μF.

The circuit details are given in Table I.

Table I

Component	*Description*
V_1, V_2	Beam tetrode type 807
R_1	Pre-set potentiometer 250 Ω, 5 watt
R_2	10K Ω, 5 watt, 20%
R_3	Potentiometer 1K Ω, 5 watt
R_4	1M Ω, 1 watt, 20%
C_1	Capacitor 0.5 μF, 1KV d.c. working
C_2	Capacitor 0.1 μF, 1 KV d.c. working
C_3	Electrolytic capacitor, 4μF, 250 V d.c. working
C_4	Electrolytic capacitor, 8μF, 600 V d.c. working
L_1	Air-gapped choke, Partridge type CF1
T_1	Intervalve Transformer, ratio 4 : 1

2.7 Watt Oscillator

This was designed as a small portable unit for demonstration purposes, and is based upon the new p-n-p junction transistors for power applications which have recently been available.

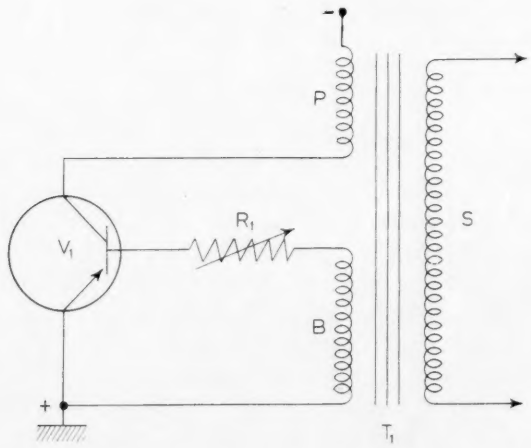

Fig. 5. A transistor power supply.

The circuit is given in Fig. 5 and all the constructional details in Table II. This is also a ringing-choke oscillator based upon an inductance using a ferrite core with a small air-gap. It can be seen that the circuit is extremely simple and consists only of three components; in spite of its simplicity however, efficiencies of the order of 70% are obtainable. The d.c. power supply of 12V is obtained from pocket torch batteries which are being used at their rating of 300mA continuous output current. R_1 is adjustable to give the correct working point of transistors having fairly wide variations in characteristics.

Frequency and waveform are variable with the load, regulation is bad, and the oscillator should not be run under no-load conditions on account of the high reverse voltage which appears across the transistor. These limitations in no way affect the use of this oscillator for electroluminescent display, where the principal requirement is for a cheap and efficient converter.

In construction, it is necessary to make sure that an efficient heat sink is provided for the transistor, and that the relative direction of the primary and base windings is correct as shown in the circuit diagram. If the load is excessive, the circuit may not be self-starting, in which case, negative bias should be applied to the base by means of a potential divider across the battery. This can be disconnected as soon as the oscillator has started.

The capacitance of a panel which is used with this power supply is of the order 500 pF, and the oscillator operates at about 1.2 Kc/s. under these conditions. The maximum output obtainable is 10mA at 270V (r.m.s.).

A 125-mW Power Supply

This was designed as a miniature piece of equipment for displaying an electroluminescent sign only $2 \times \frac{3}{4}$ inch., which could be used as a badge and carried on the lapel of a jacket.

The circuit diagram is given in Fig. 6, and the component values in Table II. The power supply is a scaled down version of the 2.7 watt oscillator, with correspondingly different circuit constants. The output is of the order 0.5mA at 270V (r.m.s.) at a

frequency of about 1.5 Kc/s. The same precautions and limitations must be observed as for the 2-watt version.

Table II

Component	*2.7 Watt Oscillator*	*125 mW Oscillator*
V_1	Pye transistor type V30/30P	Pye transistor type V10/15A
R_1	100Ω pre-set potentiometer	100Ω pre-set potentiometer
T_1	*Transformer Specification* Core: Ferroxcube 'U' core FX.1036 and Ferroxcube 'I' core FX.1067 Air gap: 1 mm. Primary: 110t, 22 s.w.g. Base: 32t 32 s.w.g. enamelled copper Secondary: 2700t 36 s.w.g. enamelled copper Insulate sections and interleave secondary	*Transformer Specification* Core: 2 Ferroxcube 'E' cores type FX.1238 Air gap: 0.5 mm. Primary: 200t, 36 s.w.g. enamelled copper Base: 80t 40 s.w.g. enamelled copper Secondary: 5000t 48 s.w.g. enamelled copper Insulate sections and interleave secondary
H.T.	12V, 300mA	6V, 30mA

PERFORMANCE CHARACTERISTICS OF PANELS

The effect of the applied voltage on the brightness of a lamp varies with the range considered. Other workers[10,11] have found that at lower field strengths up to about 10^4V/cm. the relationship is approximately an exponential function, whilst at 5×10^4 V/cm. the relationship is almost linear.

The frequency of the exciting field affects both the light output and the colour of the emitted light, particularly with phosphors which contain several activators. At low frequencies up to about 3 Kc/s. the brightness increases rapidly with frequency but at higher frequencies of the order 10 Kc/s. the lamp appears to saturate. A definite quantity of light is emitted at each electrical impulse and hence the total light output increases with frequency. It has been found that the emission of light does not exactly coincide with the changing field, *i.e.* the light intensity curve does not follow the applied voltage. There is also a slight delay in the extinction of light after the field has reached zero. The saturation effect was thus explained in terms of the persistence time of the phosphor. When the period of the applied field becomes comparable with the persistence time, the saturation effect becomes obvious to the eye. In addition to this brightness variation there is also a shift in the colour of the emitted light towards the blue end of the visible spectrum with increasing frequency.

Panels which were made early in this investigation were seen to undergo a slow deterioration by dielectric breakdown through the phosphor layer, resulting in a large number of very small black spots or pinholes. This trouble was cured, immediately a separate dielectric layer of 'Melinex' foil or melamine lacquer was introduced into the panel. Definite life tests have not been carried out on completed panels, but the types so far produced have been operated for several days without failure. One year afterwards they were found to be working, although no special precautions had been taken to seal them.

The relationship between the applied voltage and

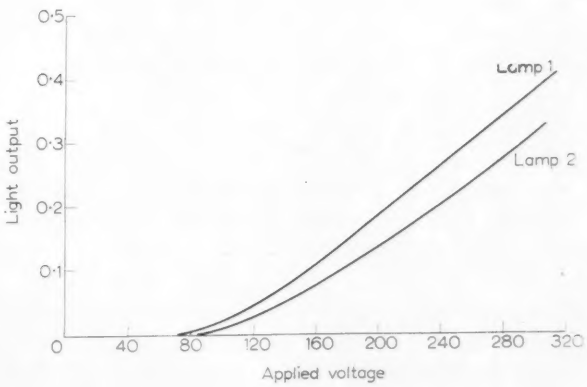

Fig. 6. The brightness of two panels is shown as a function of applied voltage. The brightness units are arbitrary, and correspond to photo-multiplier output readings.

the light output of a green phosphor was investigated using a 50 c/s. source of alternating voltage. An R.C.A. photomultiplier cell type SC223A was used to obtain a comparison of the light output of the lamp at different applied voltages up to about 300V r.m.s. The cell was placed 10 cm. from the panel and the output from the cell was measured on a moving coil microammeter. The light intensity units shown in Fig. 6 are arbitrary and are only used for comparison.

The light output in the above experiment was of a very low order, and was not measurable in photometric units with the apparatus available. Measurement of a larger panel, running at 300V and a frequency of 1.5 Kc/s., showed that the brightness of the surface was about 10 ft.-lamberts. This measurement was made with a simple photoelectric light meter.

Blue and yellow phosphors are also manufactured, but the light output for identical conditions is less than that of the green phosphor. Suitable mixtures of yellow, green and blue phosphors have been used to make a screen almost white in appearance.

FUTURE DEVELOPMENTS

Evaporation of Phosphors. Work has been in progress for several years on the evaporation of films of luminescent materials. Cathodoluminescent screens have already been made by evaporation techniques with some success[16,17]. These screens have the advantage of being almost grainless and therefore are non-scattering.

The difficulty has been in the introduction of the activator into the phosphor layer. Straightforward evaporation of a prepared cathodoluminescent zinc sulphide phosphor has not been completely successful, since the properties of the activator can only partly be restored.

The possible evaporation of an electroluminescent phosphor layer presents a unique production method for light panels and display signs, since the whole process could be carried out by vacuum deposition.

Continuous Coating. The use of a lamination technique for making the insulating layer has already been referred to. The new polyester foils such as 'Melinex' and 'Mylar' are ideal for this purpose, since they can be produced in sheets as thin as 0.00025 inch. The material does not outgas badly, and is an extremely suitable foil for coating in roll-form. The use of aluminised polyester foil thus provides an interesting production method, since it might be possible to evaporate through a mask to give a conducting aluminium pattern which could be transferred directly to the electroluminescent panel.

CONCLUSIONS

The future of electroluminescence as a source of surface light for general illumination of offices, factories and homes, is more dependent upon the characteristics of the phosphors than upon the production methods which have already been used for making panels. The efficiency and light output of the phosphor itself must be increased before the use of electroluminescence in illumination becomes an economic proposition. When this stage in the development of phosphors is reached, then serious consideration should be given to the simplified production methods arising from the use of one vacuum coating plant to perform two of the three basic operations required for making a panel. Apart from the deposition of both conducting layers, there also exists the possibility of the evaporation of the phosphor layer with the advantages that this would bring.

It can be seen that vacuum deposition can make a valuable contribution to both production and experimental work on electroluminescence. Complete experimental details have been given in this report so that other workers will be able to repeat the process.

Acknowledgements are made to Mr. P. J. Smith and Mr. R. Collins for their help in constructing the panels and power supplies. The author is grateful for the advice and encouragement given by Mr. L. Holland, Head of the Vacuum Coating Laboratory, and for permission to publish this work, granted by Mr. A. S. D. Barrett, Technical Director, Edwards High Vacuum Ltd.

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a three-electrode form of COLD CATHODE IONISATION GAUGE

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Summary

THE INTRODUCTION OF A THIRD ELECTRODE in a cold cathode gauge leads to a number of practical advantages. The characteristic change of mode, found with the conventional gauge may be eliminated; since the mode change leads to a discontinuity in the graph of ion current against pressure, this is a real merit. The sensitivity of the gauge may be changed and improved over a range of pressures which may be selected by adjustment of the voltage. In particular, the instrument may be used as a vacuum switch. In the cases which have been examined, the relative dimensions of the electrodes are important.

Sommaire

L'INTRODUCTION d'une troisième électrode dans une jauge à cathode froide conduit à un certain nombre d'avantages pratiques. Le changement de caractéristique existant dans la jauge conventionnelle est éliminé; comme ce changement conduit a une discontinuité dans la courbe 'courant ionique, pression', il y a un net advantage. La sensibilité de la jauge peut être changée et améliorée dans un domaine de pression qui peut être choisi en ajustant la tension. De plus, l'appareil peut être employé comme 'Interrupteur à vide'. Dans les cas que nous avons étudié, les dimensions relatives des éléctrodes sont important.

INTRODUCTION

THE SIMPLE COLD-CATHODE gauge of Penning[1] has many practical advantages. It is virtually indestructible and the multiplication of the ion current associated with the axial magnetic field enables the instrument to be used directly with a robust pivoted meter, instead of a galvanometer or amplifier. The characteristics of these instruments have been reviewed recently[2] and the manner in which the geometry of the electrodes affects the performance has been explored[3].

One of the merits of these ionisation gauges is that they can be used over a wide range of pressures, but there is a disadvantage in that there is an associated large variation in sensitivity. At high pressures, of the order of 10^{-2} mm.Hg there is little change of ion current with pressure while at pressures less than 10^{-4}–10^{-5} mm.Hg the variation of ion current is effectively linear with pressure. The precise limits depend upon the geometry of the gauge, the magnetic field and the applied voltage. In the intervening range there is often a discontinuity in the calibration curve, which is associated with a change in the mode of oscillation. This change of mode is accompanied by a 'hysteresis' so that the discontinuity does not occur at the same pressure when the system is being evacuated as it does when the pressure is rising. Calibration in this region is therefore unreliable.

The present purpose is to report the influence of a third electrode. The aim of the experiments was to seek a simple means, subject to external control, of shifting the position on the calibration curve of the change of mode and if possible of ensuring that it is, for practical purposes, eliminated.

* MS. received April 1957.

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EXPERIMENTAL ARRANGEMENT

The experimental gauges were all compared with a normal cold-cathode gauge fitted with a cylindrical anode[4, 2]. A convenient means of mounting a number of interchangeable electrodes is illustrated in Fig. 1 and by this means a number of experimental gauges of differing geometry were examined. The third 'focusing' electrode was insulated from the anode by glass tubing. All the electrodes could be changed easily and quickly. The electrodes were made of molybdenum sheet, nickel wire, or stainless steel gauze. Other metal parts were nickel plated to reduce sputtering. The magnetic field was provided by iron-cored coils. The anode electrode was connected through a ballast resistor[5] of 1 megohm to a power supply of nominal d.c. voltage 2,270. The potential of the third electrode was controlled by a potentiometer across a separate power supply of 6,000V and was measured by an electrostatic voltmeter. The electrical circuit is shown in Fig. 2.

The two gauges, experimental and reference, were connected directly to the pump line. Exchange of ions or electrons between the two was reduced to a minimum by using earthed metal tubing with a right-angled bend between the gauges, and by taking measurements for each gauge alternately and not simultaneously.

PERFORMANCE OF VARIOUS GAUGES

The influence of a third electrode will be discussed with reference to Fig. 3. The basic structure consists of two planar cathodes between which are mounted two ring anodes. The performance of such a gauge has already been discussed[3]. Between the twin anodes a third electrode is introduced. This electrode, referred to here as the 'focusing' electrode is also a small ring. The diameter of this ring has been kept constant in each of the four gauges which are discussed in detail below.

The first three gauges differ only in the diameter of the two anode electrodes, the dimension marked 'x', in Fig. 3; in the fourth the ring anodes are replaced by flat discs of metal gauze. Calibration curves are presented showing anode current, and current to the 'focusing' electrode as a function of pressure. In all these curves, logarithmic scales are used.

The effect of five different voltages on the third electrode was examined:—2,000V, 1,500V, 0V and —400V. Positive voltages were stabilised by manual adjustment of the potentiometer; —400V was supplied by dry batteries. These voltages refer to the voltage between the gauge cathodes and the tapping of the potentiometer. The third electrode was connected to this tapping through a ballast resistor of 660 kilohms, and therefore the actual potential of this electrode will change with the current flowing through this ballast resistor. The magnetic field was maintained at 1,000 oersteds.

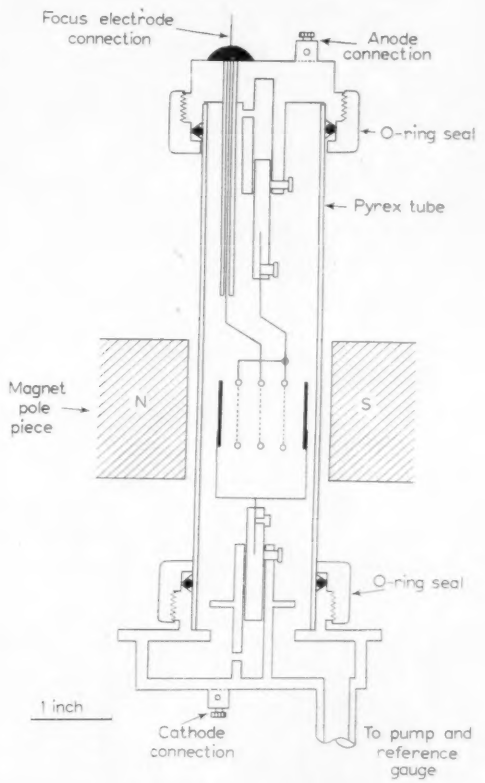

Fig. 1. Form of the experimental arrangement.

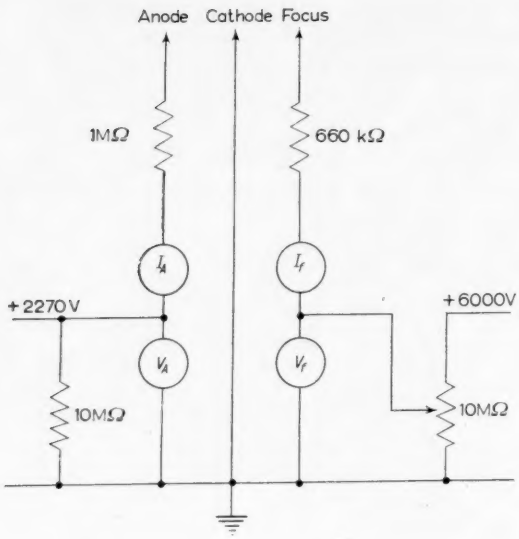

Fig. 2. Electrical circuit.

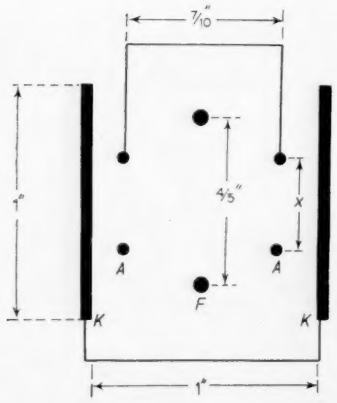

Fig. 3. Dimensions of the experimental gauges. The anode diameters "X" are given by: gauge A—1 inch (Ring anodes), gauge B—0.7 inch (Ring anodes), gauge C—0.8 inch (Ring anodes), gauge D—1 inch (Anodes of wire gauze).

Gauge A

In this gauge the ring anodes were of the same diameter as the cathodes. Fig. 4 shows the anode current, I_a, plotted against pressure for various values of V_f, the voltage applied to the third electrode. Fig. 5 shows the current, I_f, which flows to this third electrode. Large positive values of V_f ensure that electrons and negative ions flow to the third electrode. When V_f is reduced to about +400V the current reverses, as more positive ions are collected than negative particles: current flowing in this direction will be referred to as a 'negative' current.

When V_f is large and positive there are, essentially, three ring anodes and the discharge is cylindrical with a brighter ring about each of these three ring electrodes. The ionisation within the discharge is intense. As the pressure falls, the diameter of the cylindrical discharge diminishes until, at low pressures, the discharge is concentrated in a line along the axis. At backing pressures the anodes being relatively close to the cathodes define the discharge, and I_f is large since the smaller third electrode is situated in the discharge. As the pressure falls and with it the diameter of the cylindrical discharge, the smaller focusing electrode becomes a much more efficient collector of electrons and there is therefore a sharp fall in the anode current, as shown in Fig. 4. The larger the value of V_f, the more effective the focusing electrode is as a collector, and the fall in anode current therefore commences at a higher pressure. As shown in Fig. 5, the anodes then have little influence on the variation of I_f with pressure. Thus, when V_f is 1,500V, there is a sharp fall of anode current at about 1.3×10^{-3} mm Hg, and under these circumstances the gauge has effectively a single ring anode; the focusing electrode, and the calibration has little in common with that for a gauge with a double-ring anode.[3] The transfer of the function of anode to the centre ring is associated with a reduction of I_f, since the geometry ensures that the intensity of the discharge is much weakened. When V_f is 1,000V, the reduction of I_a does not occur until a pressure of about 4×10^{-4} mm.Hg is reached, and when this drop is completed at about 1×10^{-4} mm.Hg, I_f has fallen to a value lower than that measured when V_f=1,500.

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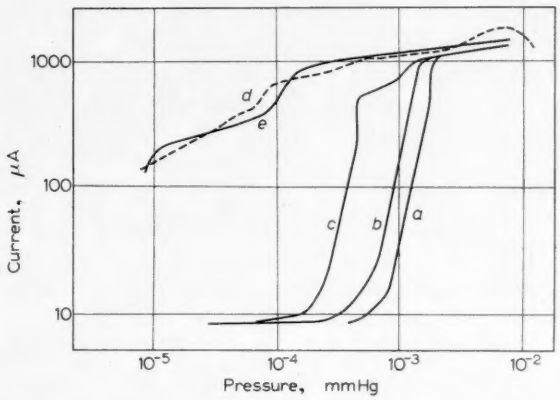


Fig. 4. *Gauge A.* Anode current as a function of pressure. Values of V_f:—(*a*) 2,000 V, (*b*) 1,500 V, (*c*) 1,000 V, (*d*) O V, (*e*)—400 V.

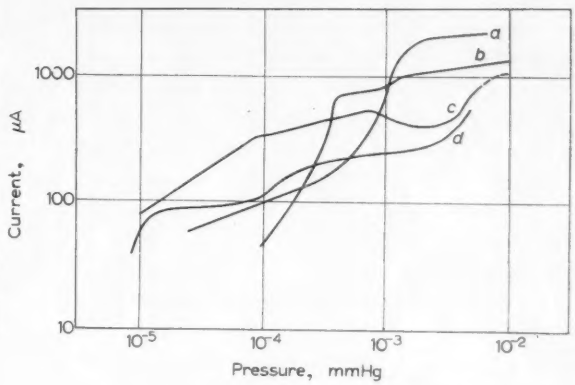


Fig. 5. *Gauge A.* Current to focusing electrode as a function of pressure. Values of V_f:—(*b*) 1,500 V, (*c*) 1,000 V, (*d*) O (Negative values of current), (*e*)—400 (Negative values of current).

Fig. 6. *Gauge B.* Anode current as a function of pressure. Values of V_f:—(*a*) 2,000 V, (*b*) 1,500 V, (*c*) 1,000 V, (*d*) O V., (*e*) —400 V, (*f*) 1,500 not stabilised. (Pressure axis staggered.)

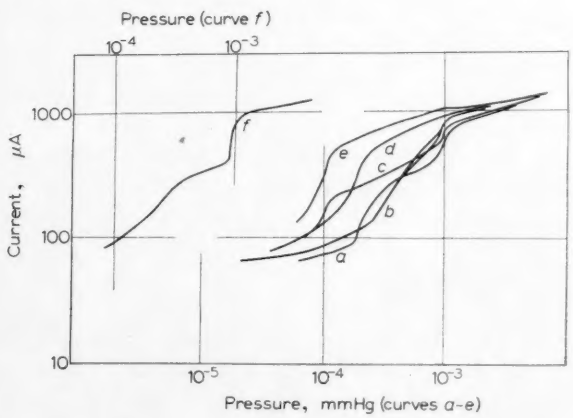
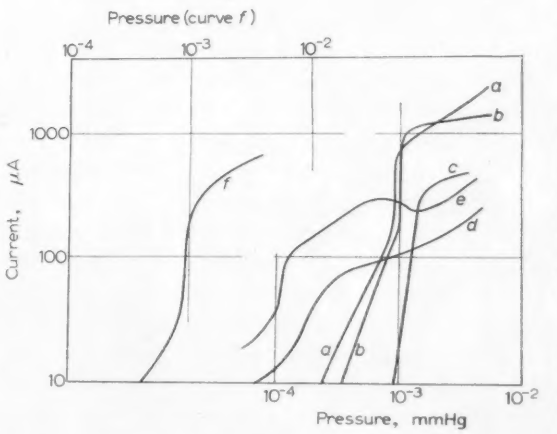


Fig. 7. *Gauge B.* Current to focusing electrode as a function of pressure. Values of V_f:—(*a*) 2,000 V, (*b*) 1,500 V, (*c*) 1,000 V, (*d*) O V, (*e*) —400 V, (*f*) 1,500 not stabilised. (Pressure axis staggered.)



This is to be expected.

When V_f is zero or negative, the gauge has, in effect, an extra cathode and I_f is negative. When V_f is 0 V or —400V, the variations of I_a with pressure are very similar save that at pressures of the order of 10^{-3} mm.Hg, I_a is more sensitive to pressure changes when the third electrode is negative. Moreover, the cathodes are at a potential of +400V relative to the third electrode and therefore attract electrons; this explains the rise in I_f at about 7×10^{-3} mm.Hg.

Gauge B

In this case the anodes were significantly smaller than the focusing electrode. The electrical and magnetic circumstances were the same as those for gauge A. Figs. 6 and 7 show the anode current, and the current flowing to the focusing electrode respectively, plotted against pressure. In general the voltages were stabilised, by adjusting the potentiometer as described above. The behaviour is much the same if the potentiometer is set with no current flowing and then not readjusted when current flows. One curve plotted in this way is shown in each of Figs. 6 and 7. The pressure scale has been staggered to avoid confusion.

Because the anodes are small compared with the focusing electrode, their roles as described for gauge A are to some extent interchanged. As the diameter of the discharge becomes smaller when the pressure is reduced, the anodes become more efficient collectors of electrons. The 'cut-off' then appears to be more marked in I_f than in I_a. The influence of V_f on I_a is most marked in the pressure range 10^{-3} to 10^{-4}mm.Hg, where the anode current is greater when V_f is 1,000V than when V_f is 1,500 or 2,000V. Presumably the third electrode has, in the first case, effectively ceased to play any part in the action of the gauge, the current to the third electrode having fallen approximately to zero. At low pressures, the anode-current characteristic of the gauge is the same for all three values of V_f. The influence of V_f on the cut-off of I_f is very marked. The smaller the value of V_f, the less effective the third electrode is, and hence cut-off occurs at a higher pressure. Thus when V_f is 2,000V, the cut-off occurs at 9×10^{-4} mm.Hg, whereas when V_f is 1,000V, it occurs at 1.3×10^{-3} mm.Hg.

As in the case of Gauge A, when V_f is zero or negative, the third electrode acts as an additional cathode. In the present case, however, not only is I_f smaller than in Gauge A, but there is an indication of a 'cut-off', a relatively rapid decrease of current in the neighbourhood of 10^{-4} mm.Hg.

Gauge C

The anodes and the focusing electrode of gauge C were all of the same diameter. The electrical and magnetic parameters were as in the previous cases. The performance is shown in Figs. 8 and 9.

With this gauge the relative influence of the anodes and the focusing electrode depends more on voltage and position than on the diameter of the ring electrodes. It should be noted that these electrodes have different ballast resistors. At high pressures, therefore, when the current is particularly influenced by the ballast, equal applied voltages may not give equal voltages on the electrodes themselves. Under some conditions the anode may be more effective as a collector than the third electrode, whereas under different conditions the position may be reversed. Thus, Figs. 8 and 9 show that when V_f is 2,000V and the pressure is 3×10^{-3} mm.Hg, I_f is approximately twice I_a, though at 8×10^{-4} mm.Hg the ballast is less important, and the two currents are of the same order; when V_f is 1,000V, I_f is less than I_a.

When V_f is not stabilised, a 'delayed ignition' effect occurs. If the gauge is switched on at a pressure below about 10^{-4}mm.Hg, the gauge does not strike, and there is no discharge until the pressure is raised above a critical value. This is indicated on the graphs in Figs. 8 and 9.

As in the previous cases, the third electrode behaved as an extra cathode when V_f is zero or —400V.

Gauge D

This gauge was the same as Gauge A, except that the anode rings were replaced by discs of stainless steel gauze. The anodes and cathodes were of the same diameter.

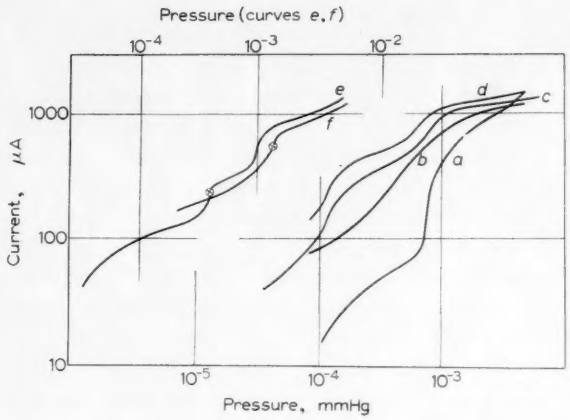


Fig. 8. *Gauge C.* Anode current as function of pressure. Values of V_f:—(*a*) 2,000 V, (*b*) 1,000 V, (*c*) O V, (*d*)—400 V, (*e*) 2,000 V not stabilised. (Pressure axis staggered), (*f*) 1,000 not stabilised. (Pressure axis staggered.) Striking point marked 'x'.

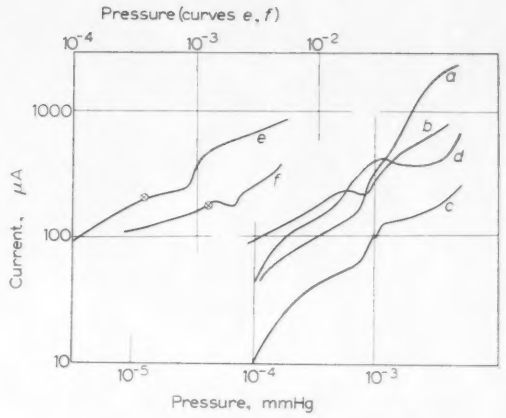


Fig. 9. *Gauge C.* Current to focusing electrode as a function of pressure. Values of V_f:—(*a*) 2,000 V, (*b*) 1,000 V, (*c*) O V (negative values of current), (*d*) —400 V (negatives values of current), (*e*) 2,000 not stabilised, pressure axis staggered. (*f*) 1,000 not stabilised, pressure axis staggered. Striking point marked 'x'.

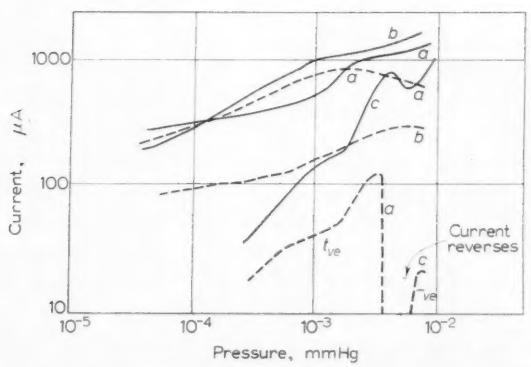


Fig. 10. *Gauge D.* Anode current and focusing electrode current as a function of pressure. (focusing electrode current as a dotted line). Values of V_f: (*a*)—400 V, (*b*) O V, (*c*) +400 V. Focusing electrode current negative, except where noted.

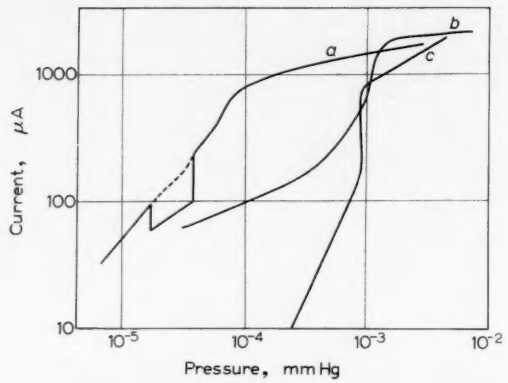


Fig. 11. *The Elimination of Mode Change.* (*a*) Gauge with single anode and no focusing electrodes. (*b*) Gauge with single anode and two large focusing ring electrodes (Arrangement A). (*c*) Gauge with single anode and two small focusing ring electrodes. (Arrangement B).

The gauze electrodes may seriously interfere with the axial oscillation of electrons between the cathodes on which the high sensitivity of these gauges depends[2]. When V_f is zero or —400V, the third electrode behaves as an additional cathode and as shown in Fig. 10 there is no very remarkable feature in the performance. There is indeed no change of mode and I_f shows a broad maximum in the region of 1.5×10^{-3}mm.Hg when V_f is —400V.

When V_f is positive the interference with electron oscillation is marked. With the customary electrical parameters the gauge would not strike. There was no discharge, if V_f exceeded 750V until it was increased to about 5,000V, when there was a discharge between the gauze and the walls of the gauge. The performance when V_f was 400V is shown in Fig. 10. The anode voltage is 2,000 and when the pressure is high the third electrode is effectively an extra cathode; V_f is negative, *i.e.* there is a considerable flow of ions to this electrode. As the pressure drops, however, the intensity of the discharge is reduced and the third electrode assumes the role of subsidiary anode and I_f changes in sign. I_f remains less than I_a, as the third electrode is at a lower potential than the anode.

THE ELIMINATION OF MODE CHANGE

In the previous sections, the gauge has been considered as having two ring anodes and a central focusing electrode. However, the simplest form of Penning gauge has but one anode between the two cathodes. It is therefore useful to compare the behaviour of such a gauge with the experimental arrangement, considering the central ring as the ordinary anode of the gauge and the outer rings as an extra electrode. The results shown in Fig. 11 are those which would be obtained if the same 2,000V power supply were used for each of the electrodes, the single anode being connected to this supply through one ballast resistor, and the twin focusing electrode connected through another similar resistor.

It is seen that the introduction of the third electrode changes the shape of the characteristic completely. The mode change is eliminated. The exact shape of the curve is clearly a function of the size of the outer ring electrodes and by suitable choice of diameters it should be possible to construct gauges having various intermediate characteristics.

CONCLUSIONS

It emerges that a cold cathode ionisation gauge, having three coaxial ring electrodes between the cathodes, may exhibit a number of different characteristic curves of pressure plotted against ion current. Either the central ring electrode or the two outer ring electrodes, strapped together, may be used as the anode with the other acting as a focusing electrode. A single instrument may be used as a vacuum switch with a sharp cut-off, as a conventional cold-cathode gauge of modest sensitivity or as a gauge of relatively low sensitivity over a wide range of pressures; the performance is determined by the voltages applied to the electrodes. Such gauges may be operated in such a manner that they are free from the discontinuous changes in mode which are such an annoying feature of the common simpler form of gauge.

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HIGH VACUUM GAS-LEAK VALVES

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Summary

THIS PAPER gives a brief survey of a variety of types of valves suitable for the controlled admission of gases into a high vacuum system. The principal features of this type of valve are discussed first and followed by a more detailed description of the following: Needle valves, slit valves, valve fitted with porous plugs, valves operating by means of rubber plugs pressed into grooves machined into the housing, valves admitting an intermittent flow of gas, and valves, the action of which depends on the effect of temperature variations in the construction materials.

Sommaire

CET ARTICLE à pour but de donner un aperçu bref de différentes constructions de robinets à microfuites ajustables au moyen desquels on peut admettre un gas dans un système à vide élevé. On discute tout d'abord les characteristiques générales de ces robinets. Ensuite on décrit plus détaillé les différents types actuellement employés: des robinets d'aiguille, de fente, munis d'un élément poreux, munis des anneaux en caoutchouc pressés dans les rainures, robinets à admission périodique et enfin des robinets dont le fonctionnement dépend sur un change de temperature des materiaux de construction.

INTRODUCTION

IT IS FREQUENTLY necessary to arrange for the admission of a controlled amount of gas into a highly evacuated system such as the ion source of a mass spectrometer. Other examples are c.r. and discharge tubes in production, the testing of the speed of vacuum pumps, the study of adsorption phenomena and all experimental work where it is an important requirement that the pressure in a continuously pumped system remains constant for the duration of the work. These are typical instances where valves of the kind described in this article, *i.e.* the so-called gas-leak valves are employed.

It is the object of this article to give a review of existing types of gas-leak valves and discuss in particular, principal design features and specific performance characteristics. The admission of gas to a vacuum system by the mechanism of diffusion such as the admission of hydrogen through palladium[1,2], nickel[3,4], etc. is outside the scope of this article and is only mentioned here to complete the account of the means available for the purpose.

THE BASIC FEATURES OF GAS-LEAK VALVES

One of the basic characteristics of a gas-leak valve is the range of values of the vacuum conductance C covered whereby C is defined by the equation

$$C_1 = \frac{Q}{P_1 - P_2} \quad (l.\,sec^{-1}) \qquad (1)$$

Where Q is the amount of gas passing through the valve, P_2 is the pressure of the gas leaving the valve and P_1 is the pressure of the gas entering the valve.

Normally the gas-leak valves are employed to admit gas which is at atmospheric pressure. Therefore the pressure difference P_1–P_2 in Equ. 1 is very nearly constant in all cases. This is the reason why the range of values within which it is possible to change

* MS. received December 1957.

the flow of gas Q passing through the valve, is frequently used as the essential criterion in assessing individual designs. The gas flow Q is defined as the volume V^1 of the gas which has passed through the valve in unit time at a pressure P and this is given by

$$Q = PV^1 \ (\mu l.\ sec^{-1})$$

Another basic distinguishing factor is the range of pressures, for which the valve can be used. The merits of a valve depend on the accuracy with which predetermined values of conductance or gas flow can be reproduced.

In certain types of gas-leak valves a fluid such as mercury is used to control the passage of the gas and this requires the provision of a trap for the condensation of the mercury vapour. Thus the need for a cold trap is another feature characterising the design of a valve. It should be added here that, unless mercury vapours originating from other sources may be present in the system at the same time, this type of gas-leak valve offers no particular advantage.

The specific uses of a valve depend very largely on the material from which it is made. In the case of a glass vacuum system, a glass valve will be used and in the case of a metal vacuum system, a metal valve will be used. The construction material of the valve decides whether the valve may be outgassed.

There are of course other factors which count in the assessment of a valve such as sturdiness of construction, simplicity of operation and cost of manufacture.

AVAILABLE TYPES OF GAS-LEAK VALVES

The first gas-leak valves which became available were of the needle-valve type. These valves are normally of an all metal construction and work on the principle of a needle (with a conical tip) made from comparatively hard material being forced into a seat made from comparatively soft material. The operation of the valve is controlled by means of a screw joined to the needle. Sometimes a differential screw is used instead of a standard-size thread. In a particularly efficient design of needle valve the position of the needle in the seat is closely controlled[5] and hence the conductance can be adjusted with great precision. Generally speaking, the needle valve has the drawback that accurate reproduction of values of gas conductance is difficult owing to the deformation of the valve seat at an early stage of the service life of the valve.

Effective air-tightness of the valve is usually obtained by using rubber components. This prevents the valve from being outgassed with success. Many modern designs are fitted with a cone made from silver carrying a tip made from hard material in place of the needle, the tip fitting into a seat made from equally hard material.[6] In such instances satisfactory air-tightness is ensured by the provision of an elastic membrane joined tightly to both the cone and the body of the valve, see Fig. 1. Such valves stand up to outgassing treatment involving a temperature of several 100°C.

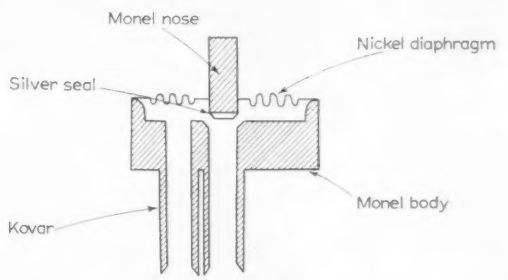

Fig. 1. Ultra-high vacuum all-metal valve designed by Bills and Allen.

Another type of gas-leak valve is the slit valve. An early design of this kind of valve, proposed by J. J. Hopfield, has a vertical slit at the closed end of a capillary tube. The width of the slit can be varied by shaping the tubes as shown in Fig. 2a and moving the tubes as indicated by the arrows.

This design affords a good control of the quantities of gas admitted but the range of conductances is comparatively small and operation is delicate.[7] Fig. 2b shows a valve with a longitudinal slit, designed by Kunzl and Slavik[8]. The valve has many favourable characteristics. The operative dimensions of the slit

may be varied by raising or lowering the level of the mercury in the enveloping tube in a simple manner, as shown in the illustration. In this valve predetermined values of conductance can be reproduced without difficulty but the valve cannot be outgassed and a cold trap is required to condense the mercury vapours. An improved design is described by Amoignon *et al.*[12]

The third group of valves to be discussed in this article is the type fitted with plugs of porous material. Fig. 3 shows a design described in 1933[9]. The valve is made of glass. At the lower end of the tube the porous component can be seen shaped in the form of a cone. As the plunger actuated by an electromagnet moves downward, the level of the mercury in the tube rises and progressively covers the surface of the cone so that the conductance through the cone is steadily reduced. When the cone is fully submerged the valve is effectively closed. A similar construction was employed by Glaister[10]. Here, a disc of sintered glass is substituted for the cone, see Fig. 3b. Valves of this kind are simple in construction and easy to manipulate but require mercury for their operation. Also, action is protracted owing to the fact that diffusion of gases through the porous component is slow.

The representative design of another family of valves is shown in Fig. 4a and was designed by M. H. Mueller and R. B. Bilinski[11]. In this type of valve a series of rubber plugs with metal discs between them serving as distance pieces are situated in the axis of a cylindrical envelope into which fine grooves have been machined. The gas conductance of the valve is varied by exercising pressure on this rubber-metal disc system *via* the plunger shown in the drawing. The latter can be moved by rotating a threaded sleeve. The sleeve motion is effected through a differential gear and can be accurately controlled within very fine limits. Under the compression thus obtained the rubber plugs expand into the grooves. The degree of leakage in the valve therefore depends on the pressure exercised by means of the plunger. The design of a valve operating on a very similar principle has been suggested by Amoignon *et al.*[12], see Fig. 4b. Here the grooved component is arranged in the centre and has the form of a metal cylinder. Two rubber 'O' rings are placed around the grooved cylinder and can be compressed under the action of a screw driving the stainless steel tubes indicated in the illustration. The effect is the same as that described in the previous model but the latter design has the advantage that the grooved component is easily disassembled and as a result can be exchanged without difficulty against a cylinder with different groove dimensions. Thus the range of values of gas conductance of the valve is larger than in the previous design. This family of valves is well suited for application in conjunction with metal vacuum systems. The valves are robust and can be easily manipulated; however, they cannot be outgassed.

C. H. Bachman[13] and A. J. Stinnett[14] constructed valves which provide for the intermittent admission of small quantities of gas to the system. This type of valve forms another distinct group within the family of gas-leak valves. Bachman's design is shown in Fig. 5a and essentially consists of a cylinder with a few grooves of substantial dimensions on its circumference. The cylinder can be rotated and as a result, in a particular position, each groove in its turn will effect a connection between the gas reservoir and vacuum system. In this way it is possible to admit well-determined quantities of gas into the vacuum system. The conductance of the valve is changed by altering the revolutions of the cylinder. Such a valve is useful if the system in question is of a large volume but where the system is of moderate volume the valve has the disadvantage of producing appreciable pressure changes in the system. Stinnett's design, an all metal construction, consists essentially of a vibrating needle as shown in Fig. 5b. The needle forms an integral part of a rod extending from a plunger situated in the axis of a solenoid which is supplied by alternating current. The amplitude of the oscillations of the needle and therefore the magnitude of the conductance can be varied by changing the position of an adjustment screw. This type of valve can be readily outgassed.

A very simple design of valve has been proposed by R. K. Smither[15]. In this case the valve consists of a thin capillary glass tube carrying a coil of resistance wire on the outside. On heating the tube by the

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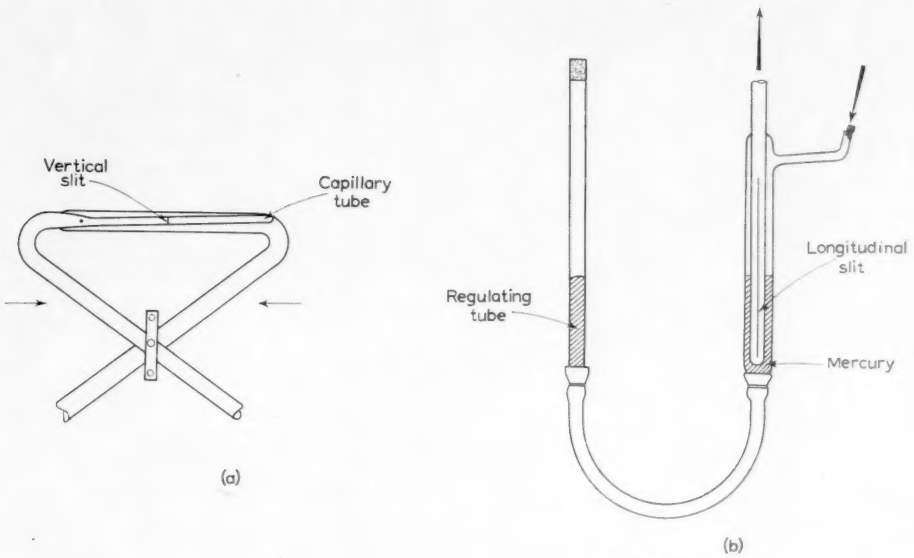
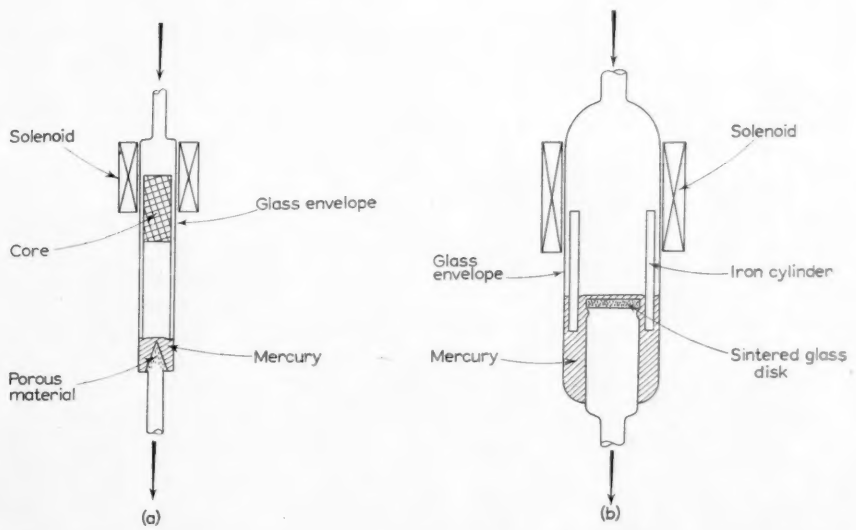


Fig. 2. Slit valves: (*a*) Hopfield's design; (*b*) Kunzl and Slavik design.

Fig. 3. Valves fitted with a component of porous material. (*a*) Design by Köhler and Rompe; (*b*) design by Glaister.



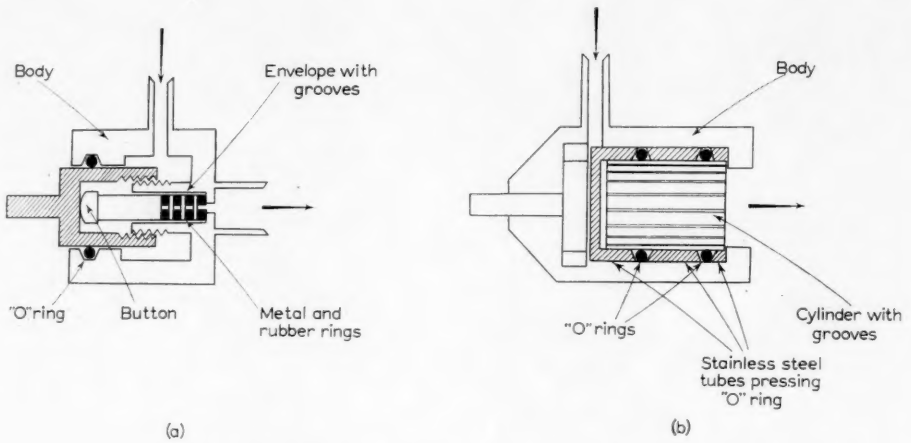
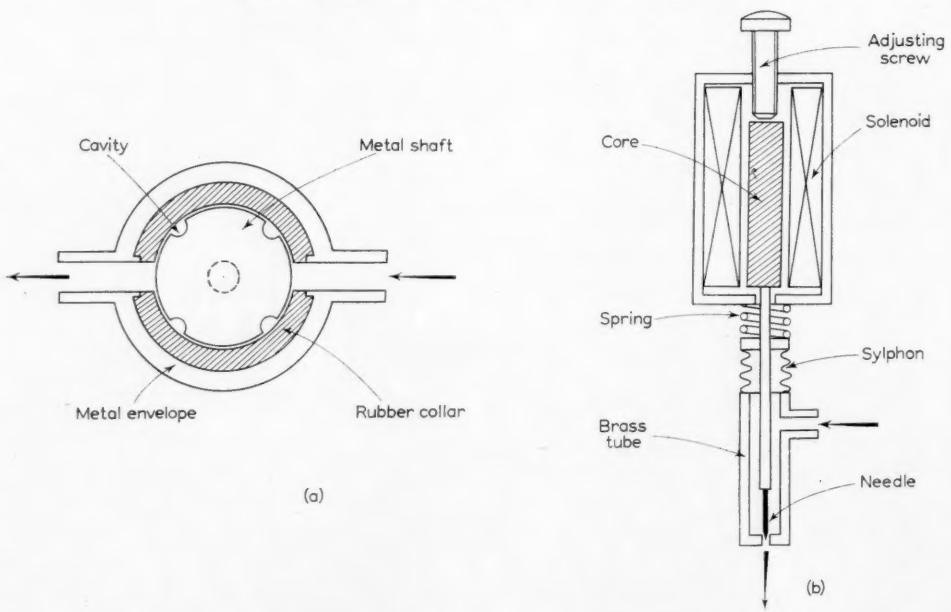


Fig. 4. Valves fitted with rubber plugs expanding into grooves machined into the envelope. (*a*) Design by Mueller and Bilinski; (*b*) design by Amoignon *et. al.*

Fig. 5. Valves designed for intermittent admission of gas. (*a*) Bachman's design; (*b*) Stinnett's design.



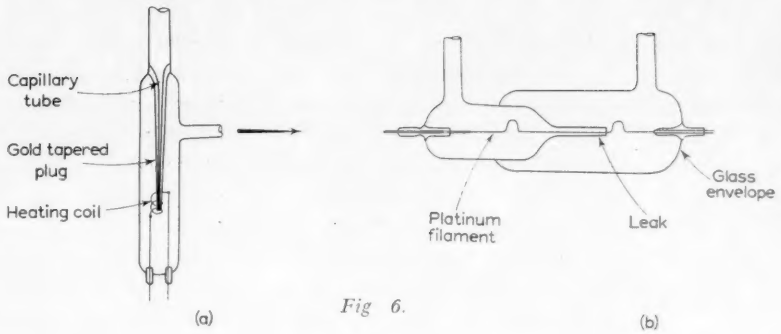


Fig. 6. Valves forming a gas leakage path as a result of the difference of the temperature expansion coefficient of the materials. (*a*) Nester's design; (*b*) Martin's design.

Fig 6.

resistance winding the gas conductance of the valve will vary in accordance with the equation for the flow of gas through tubes of circular cross-section. The range of the conductance values obtainable in the valve depends simply on the maximum diameter to which the capillary tube will expand under the effect of the heat supplied. However, a serious drawback inherent in this design, is the fact that the valve cannot be fully closed. On the other hand, the valve has no metal parts and this makes it particularly useful in connection with the manipulation of reactive gases. Another design of exceptional simplicity has been described by Morrison[16]. Here a glass slide ground to very fine limits is made to travel over a

Table 1.

Valve Designer	*Operational Principle*	*Flow Range μl/sec.*	*Sealing Pressure mm. Hg*	*Construction Material*	*Can be Outgassed*	*Requires Trap*
1	2	3	4	5	6	7
Babelay–Smith	Needle.	$0–6 \times 10^{-2}$	10^{-5}	metal	—	—
Bills–Allen	ditto.	—	10^{-14}	metal	×	—
Kunzl–Slavik	slit.	$0–3.8 \times 10^{2}$	10^{-8}*	glass and mercury as sealing fluid	—	×
Glaister	Admission through porous material.	$0–2.5 \times 10^{3}$	10^{-8}*	ditto	—	×
Mueller–Bilinski	Sealing through rubber plugs forced into grooves of the envelope.	—	10^{-5}*	metal and rubber sealing components	—	—
Amoignon *et al.*	ditto.	0–4	10^{-5}	metal and rubber	—	—
Bachman	Intermittent admission.	—	10^{-4}	metal and rubber	—	—
Stinnett	ditto.	—	10^{-5}	metal	×	—
Smither	Admission varied through expansion of the material caused by heat.	3–200	5×10^{-2}	glass	×	—
Morrison	Admission through holes of different diameter.	$2.1–760 \times 10^{3}$	10^{-5}	glass and metal	×	—
Nester	Admission as a result of the difference of temperature expansion coefficient of two materials.	—	10^{-7}	glass and metal	×	—
Martin	ditto.	0–30	10^{-8}*	glass and metal	×	—

*—values not actually mentioned in the respective publication but deduced from construction data.

metal plate placed in the path of the gas leak. As the slide is moved forward it progressively covers up holes of different diameters. This design allows for air-tight closure of the valve but any change of conductance is necessarily stepwise, and not continuous. Valves of this kind have been used in experiments concerned with absorption capacity of getters.

Finally, a group of valves should be mentioned, the operation of which is based on the expansion or contraction of a small gap originating between materials having different temperature expansion coefficients as a result of temperature changes. A typical example of a valve of this kind is shown in Fig. 6 and was designed by Nester[17]. As shown in the drawing the leak tube ends in a point closed by a small amount of gold. In the neighbourhood of the gold point is a heater coil. While the temperature is kept at about 300°C, the valve is fully closed. When the temperature is lowered a gap forms between the glass and the gold through which gas may pass. This valve is very useful for the automatic control of pressure in vacuum systems. Another design shown in Fig. 6b is suggested by Martin. In this valve the materials in question are platinum and glass. The platinum loop is heated by electric current and this causes the formation of a leakage path between the metal and the glass. The size of the leak depends on the temperature attained in the loop. The valve can be tightly closed and the range of conductance values can be altered without having to dismantle the valve itself. All that is needed is to overheat the capillary tube to the softening temperature of the glass and admit gas of high pressure to one or the other end of the valve, as a result of which the internal diameter of the capillary tube will expand or contract respectively.

Table I summarises the essential characteristics of all the valves discussed in this article.

CONCLUSIONS

Gas-leak valves for laboratory purposes are very delicate devices. With the exception of certain types of needle valves all the valves discussed in this article have been fabricated by various research workers to meet specific needs and functional requirements for special experimental arrangements. Information on these valves was made available mostly in the form of notes published in a great variety of scientific magazines. As a result the same type of valve has been developed over and over again by workers unaware of the fact that details of the design in question are already available. As the matter stands today, there are several types of gas-leak valves available for use in a variety of applications.

It is hoped that this article will benefit the research worker and save him valuable time spent in the past on the design of a gas-leak valve to suit his equipment.

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LETTERS TO THE EDITOR

A Vacuum Desiccator

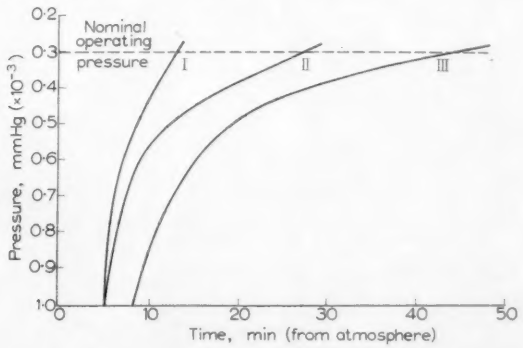
The advantage of removing occluded gas and water vapour which is present in photographic plates and films prior to subjecting these materials to high vacuum conditions is well known among users of the electron microscope (*e.g.* a decrease in general pumping-time); in some cases manufacturers provide a means of degassing, but this is the exception rather than the rule. The following, therefore, describes a simple and efficient vacuum desiccator which can be easily constructed where workshop facilities are available.

The vacuum desiccator comprises three main parts: the pumping chamber and two standard items, a two-stage rotary pump (Metro-Vickers type D.R.1), and a moisture trap (Edwards type 5A). The chamber, see Fig. 1, is mounted above the moisture trap which is connected to the rotary pump; this arrangement avoids the use of an additional pumping line and saves considerable workshop time in construction. The body of the chamber is constructed from a metal cylinder 23 cm. long with an internal diameter of 11.4 cm.; this size of chamber was specifically designed to hold photographic plate cassettes of the type used on the E.M.3 electron microscope (Metropolitan-Vickers) and is capable of accommodating up to ten cassettes. The chamber is flanged at the top and bottom; the top flange supports a hinged lid, which makes a vacuum-tight joint with the flange by trapping a rubber 'O'-ring recessed into the underside of the lid.

The bottom flange of the chamber seals directly on to an 'O'-ring around the top of the moisture trap and is secured by the clamping ring normally intended to hold a glass plate. An internal flange at the bottom of the chamber carries, on its underside, a hinged flap valve complete with 'O'-ring seal; this flap valve may be closed to isolate the chamber from the rest of the system by a cam situated below the valve on the opposite side to the hinge. The shaft to which the cam is attached passes through a vacuum seal (Wilson-type) in the side of the moisture trap and is operated by an external control. In order to give sufficient clearance for the flap valve in the open position the upper desiccant tray of the moisture trap is removed and the central pumping stem appropriately shor-

Fig. 1. Chamber of the vacuum desiccator specially designed for the drying of photographic plates.

Fig. 2. Pumping-down times of E.M.3 electron microscope. (*i*) No photographic plates present. (*ii*) Photographic plates in their cassettes—pre-pumped for 20 mins. (*iii*) Photographic plates in their cassettes with no pre-pumping.



tened. Two steel supports are fixed between the moisture trap and rotary pump to avoid the risk of overstraining the vacuum coupling.

The chamber can be left under vacuum conditions (*e.g.* overnight) by closing the flap valve, but air is admitted to the pump in the usual way by the R.S.2. valve fitted to the moisture trap. When cassettes are required after degassing, air is admitted to the chamber by another R.S.2 valve provided on the top lid of the chamber. The graph in Fig. 2 relates the time involved in reaching nominal operating pressure (0.3μ Hg) for the E.M.3 electron microscope. Tests were carried out from atmosphere to 0.3×10^{-3} mm. Hg. The pressure measurements were made with a cold-cathode type vacuum gauge and fresh desiccant was used both in the backing line of the microscope and in the pumping chamber before the readings were taken. It was found that when pre-pumped plates were inserted in the microscope the time required to reach a pressure of 0.3μ Hg was only one-half of that needed when wet plates were used.

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4th April, 1957.

A New Evaporation Source for Vacuum Deposition of Aluminium

A CONSIDERABLE NUMBER of different types of evaporation sources to be used in connection with the vacuum deposition of aluminium in large quantities have been described recently[1]. To quote two examples, Holland and Cutter designed a source which consisted essentially of a stranded tungsten spiral into which an aluminium wire was fed at an intermittent rate and Bancroft reported on a method employing high frequency power for heating purposes. However, the former requires a rather complicated feeding mechanism and the latter suffers from the considerable expense involved in the provision of a high frequency power generator.

Below, we report on a novel design of evaporation source to suit the same purposes as those quoted before but being of comparatively simple construction.

In vacuum evaporation the aluminium must be raised to a temperature of about 1,300°C. There are no metals or alloys which are known to be completely immune to the attack by molten aluminium and could therefore be used as construction material for the source[2]. As far as our experience goes, graphite too, reacts with molten aluminium at high temperature forming a carbide which not only causes the graphite crucible to disintegrate but in addition has an adverse effect on the quality of the aluminium deposit. Crucibles made from ceramic materials such as highly sintered alumina, zirconia etc., are known to resist the attack of molten aluminium satisfactorily, but they are of little use in connection with the construction of sources because of their poor resistance to thermal shock.

It has been noted[3] that at the required operational temperature aluminium is a highly mobile liquid which readily wets the walls and tends to overflow the crucible.

The authors have found that bars of titanium carbide (TiC), if prepared with due care, show a good resistance to liquid aluminium at high temperature. Titanium carbide belongs to the class of the so called 'hard metals'[4]. Its thermal electrical conductivity values are of the order of magnitude applicable to pure metals. It further has a high modulus of elasticity, a high melting point and a correspondingly high resistance at elevated temperatures. Bars of titanium carbide can be conveniently prepared by a powder metallurgical process but owing to the poor workability of the material generally it is difficult to produce titanium carbide components of a comparatively complicated shape.

The new evaporation source is designed to suit the characteristics of the titanium carbide material and the properties of the liquid aluminium described before. Fig. 1 gives a diagrammatic view of the new source. A and A^1 are the titanium carbide bars, B represents the aluminium tips and C is a crucible made from ordinary alumina. The bars A and A^1 are placed in the crucible so that their ends are in contact with each other and electric current can be passed through them. Initially while the resistance of the surfaces of the bars in contact with each other is high the heating effect is concentrated in the area of the bars. This causes the aluminium near the two end surfaces of the bars to melt and wet the bars. As a result the temperature near these points in the bars drops and the zone of maximum temperature in the bar is moving upwards. Accordingly the molten aluminium creeps upwards along the sides of the bars A and A^1 and evaporates when it reaches the zone of maximum temperature in the bar. In this manner almost the whole of the aluminium content of the crucible is being evaporated. At the same time the temperature of the molten aluminium in the crucible itself is not high enough to attack the construction material of the crucible and wetting phenomena of the kind discussed above leading to an overflow could not be observed. This is the reason why ordinary

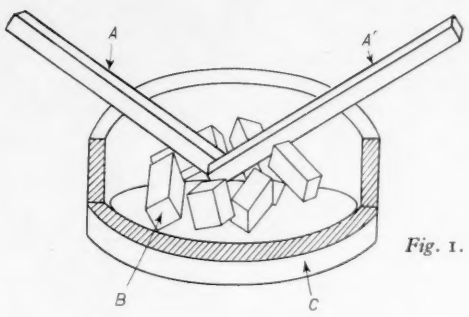


Fig. 1.

Fig. 1. Diagram of the new source.

Fig. 2. Apparatus constructed for testing the new evaporation source. *Legend*: A.A.[1]—TiC bars, C—crucible, S—shutter, G—glass cylinder, R—rotating disc for measuring rate of deposition, T—Al cylinder, M—mirror for watching.

Fig. 3. Configuration of the pin hole camera (*a*) and a figure obtained (*b*). *Legend*: A.A[1].—TiC bars, P—pin-hole, C—crucible, G—glass plate.

Fig. 4. Relation between film thickness deposited in a second and input electric power.

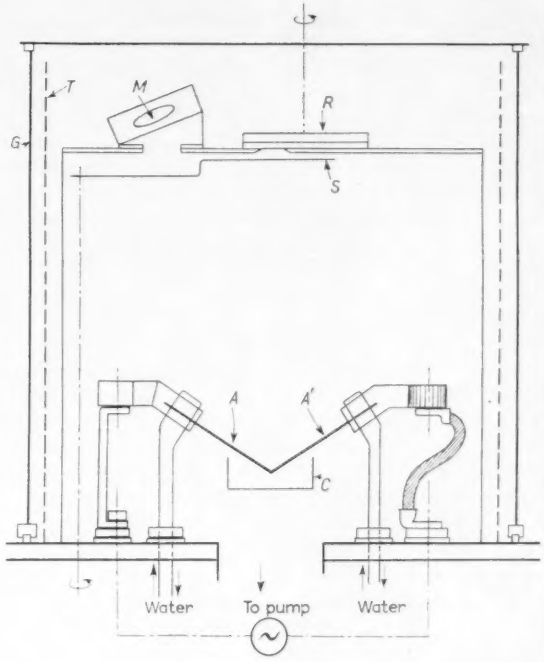


Fig. 2.

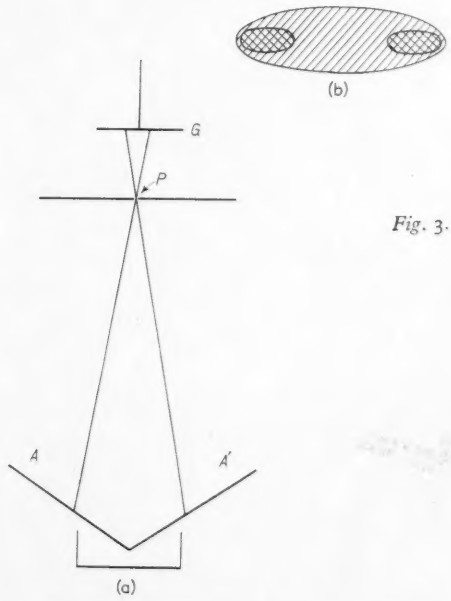
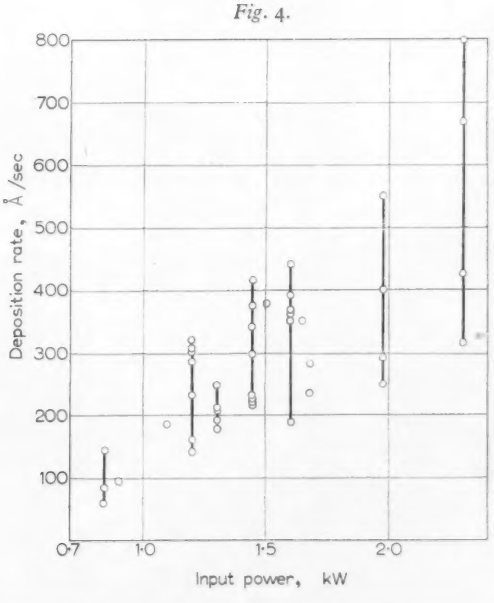


Fig. 3.

Fig. 4.



alumina crucibles can be used as containers for the molten aluminium in this source design.

Fig. 2 shows an apparatus which was specially developed for the purpose of testing the performance of the evaporation source just described. The titanium carbide bars used in that instance had the dimension of $56 \times 5 \times 5$mm. The rate of evaporation was determined by measuring the thickness of the aluminium deposit obtained in a unit time on a substrate at a given distance from the source. The distance was 18cm. The area of the maximum temperature in the bar *i.e.* the zone at which vaporisation occurred was determined by means of a pin-hole camera. The arrangement of that instrument and the type of record obtained from it are shown in Fig. 3. In the particular case the record demonstrates that the maximum evaporation took place at a point in the bars about 25mm. away from their lower ends. Evaporation from the pool of molten aluminium in the crucible is negligibly small. The thicknesses of the aluminium deposits were measured with the help of Fizeau's interference fringe technique.

Fig. 4 shows the relation between rate of deposition and source input. The values obtained were rather dispersed, but the mean values seem to give a straight line. Fig. 4 demonstrates that the efficiency of the new evaporation source increases with the power input.

For the purposes of the test the crucible was charged with 10g. of aluminium at a time and evaporation could be carried out without interruption for 20 or 30 minutes. Evaporation came to an end when the aluminium placed into the crucible was consumed or in the event of the titanium carbide bars breaking. The causes for these breakages have not been fully ascertained yet and if this can be overcome the life of the evaporation source can be expected to be considerably longer.

In place of TiC, other hard metals may be used such as SiC and SrB. The latter two materials may prove to be more suitable in some respects.

Summing up we would like to say that titanium carbide sources facilitate the deposition of large quantities of aluminium. The new evaporation source can be employed with advantage in roll-coating operations.

We are greatly indebted to Dr. Haruo Kubota and Ryuji Fukuda of the Sumitomo Electric Industry who kindly supplied us with the titanium carbide bars for our experiments. Details of the preparation of the bars will be published elsewhere by that company.

We are grateful to Dr. Tatsuo Takatsuki of Nikku Vacuum Industry for the encouragement he has given us throughout this work.

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3rd December, 1957.

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MICROSCOPE HOT STAGES

IT IS SOMETIMES DESIRABLE to examine small objects under the microscope at elevated temperatures. The size of the stage that can be used depends upon the N/A of the objective. For high magnifications the

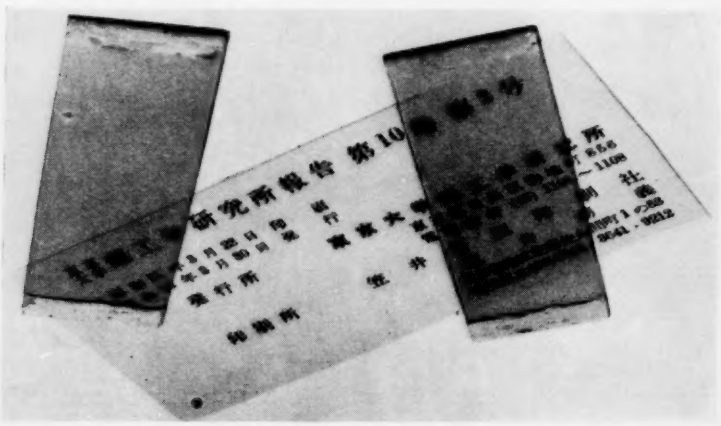

Fig. 1. Two coated slides arranged to show the small loss of light in transmission.

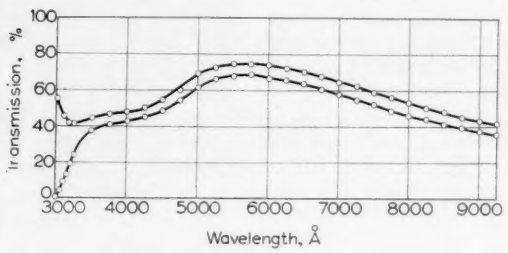

Fig. 2.

Fig. 2. Per cent of transmission *versus* wave-length of gold conducting layer shown in the upper curve and the corresponding values of the glass heating slide are shown in the lower curve.

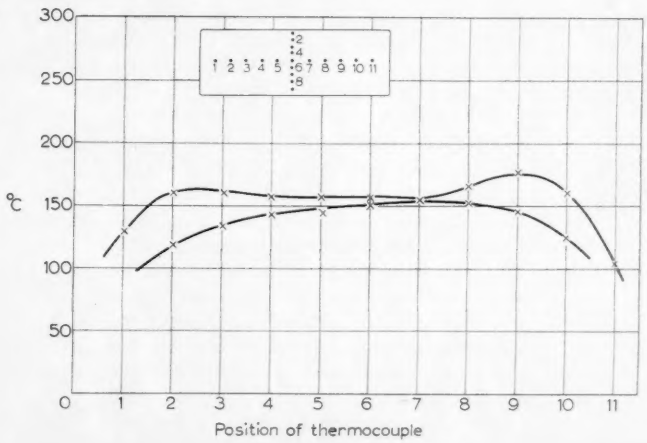

Fig. 3.

Fig. 3. Temperature distribution along the slide and across the slide at 10 V. The points of measurement are shown in the insert.

Fig. 4. Gives a view of the arrangement on the microscope.

Fig. 4a. A detailed view of the assembled microscope stage.

Fig. 6. Detailed view of a small furnace employed in the study of crystal changes at high temperatures.

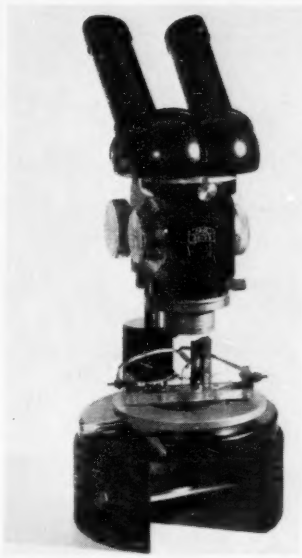

Fig 4.

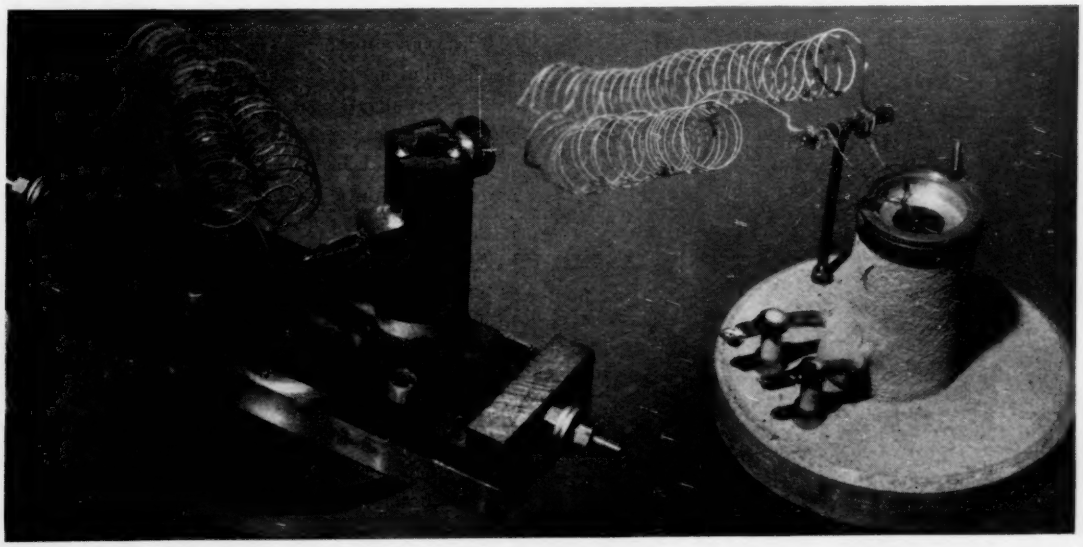

Fig. 4a.

Fig. 6.

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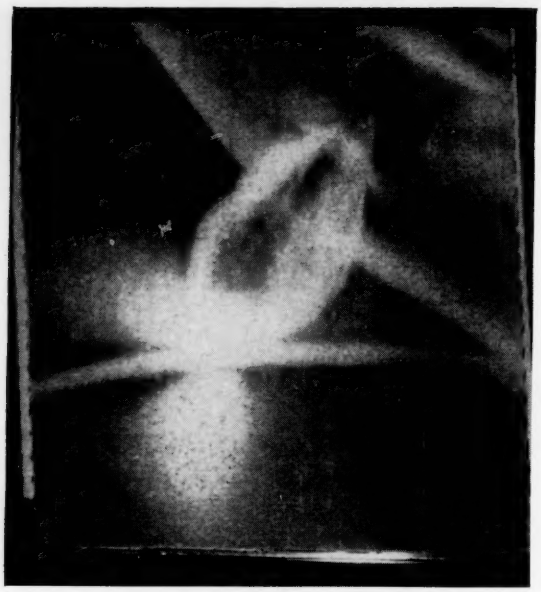

Figs. 5 and 5a. Different stages in the burning of crystals of mercury fulminate at 100°C. (Magnification × 80.)

working distance between the lens and the object being quite small, special types of hot stages must be made. In a number of investigations being made in the laboratory[1] it has been necessary to follow physical and chemical changes taking place in single crystals of a number of materials at elevated temperatures. In some investigations a transparent hot stage is essential.

A simple hot stage has been made from 3 inch × 1 inch microscope slides prepared by coating one side with an extremely thin film of gold which is covered by an even thinner protecting film of bismuth-oxide[2]. A strip of indium across each end facilitates electrical contact so that current may be passed from end to end, to warm up the glass slide. Temperatures up to 150°C have been maintained for fairly long periods; above this temperature there is a risk of failure due to breakdown of the circuit between the indium contact and the gold film. For higher temperatures, in the region of 350°C, silver contacts were made by fusing a narrow strip of silver (Johnson and Matthey silvering pamphlet 3473) across the ends of the slides before coating with gold. The upper limit of temperature using silver contacts has not been ascertained, but runs up to 350°C have been made. In Fig. 1 two coated slides are arranged to show the small loss of light in transmission and this is shown quantitatively in Fig. 2 where the percentage transmission of light is plotted as a function of wave length. The temperature distribution along and across the slide is given in Fig. 3. The arrangement on the microscope, see Fig. 4, shows the coated slide resting upon a pair of metal contacts which have been suitably mounted on a bakelite base shaped to clip into the microscope stage, detailed in Fig. 4a. The hot junction of a thermocouple is held against the upper uncoated side of the slide by a swivelling and adjustable glass probe which also serves to press the indium contacts of the heating slide against the metal contact blocks.

The coated slides have been used in the study of the decomposition of small crystals over a range of temperatures. They have also been used to investigate the effect of temperature on the high-speed burning of single crystals of a number of materials[3] the same arrangement as in Fig. 4 is used in transmission on a high-speed lenticular plate camera. The photographs in Fig. 5 show different stages in the burning of crystals of mercury fulminate at 100°C (× 80). The actual size of the crystals used is quite small and their thickness is only a fraction of a millimetre. A small furnace has also been constructed, see Fig. 6, which has been successfully used in combination with a stereomicroscope to study changes in crystals when heated to temperatures as high as 450°C. It is made of a hollow core of silver around which a nickel-

chrome heating element is wound and cemented in place. The top and bottom of the heating chamber is made from microscope cover glasses so that transmitted light may be used. The height of this furnace is an inconvenience at low powers because the microscope body tube cannot be racked high enough to bring the object into focus. The heat, rising from the furnace or coated slide, can damage the microscope objective situated immediately above and a blower is mounted nearby directing a draught of cold air across the objective's lower surface. The arrangement as shewn is simple and improvements can readily be made. It is however suitable for investigations of the type mentioned earlier.

A study of the physical and chemical changes which take place during thermal and photochemical decomposition of a number of materials such as the inorganic azides and fulminates is at present being made and both stages of the types described have been valuable in preliminary studies of such problems.

I thank Dr. F. P. Bowden, F.R.S. for his interest in this work.

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1st March, 1957.

[1] Laboratory for the Physics and Chemistry of Surfaces, Department of Physical Chemistry, Cambridge.
[2] This technique was originally developed at the National Physical Laboratory, Teddington.
[3] See, for example: *The Burning and Explosion of Single Crystals* by B. L. Evans and A. D. Yoffe, *Proc. Roy. Soc.*, **238a** 325, (1957).

BOOK REVIEWS

Physik Dünner Schichten. By H. Mayer. Vols. I and II published 1950 and 1955 respectively, 326 pages and 390 pages. (Wissenschaftliche Verlagsgesellschaft m.b.H., Stuttgart).

"*. . . many points imagined in continuous contact do not constitute the line, and as a consequence many lines in continuous contact as regards their sides do not make a surface, nor do many surfaces in continuous contact make a body, because among us bodies are not formed of incorporeal things.*" Thus passes Leonardo da Vinci from the abstract to reality, a path we may follow today by way of that corporeal system known as *thin solid films* where the height of the system is measured in equivalent mono-molecular layers or units of one hundredth millionth of a centimetre.

Using vacuum deposition techniques thin films may be prepared ranging from mono-atomic layers to those of optical interference thickness. The properties of such films have been investigated from almost every standpoint over the last seventy years until now the resultant annual output of scientific papers has grown beyond the reading capacity of a single person. Not only has our knowledge of the physics of solids and surfaces been extended by this intensive work but we also have the many useful applications of thin films in optics, electronics and research, etc. The subject has not developed however, without theoretical difficulties. One of the commonest stumbling blocks has been the unquestionable application of the *concept* of an ideal thin film system consisting of a smoothly bounded layer of a homogeneous and pure material to the products of the vacuum evaporation of sputtering plant, which, depending on their mode of growth, may be extremely granular and impure.

Professor Mayer has set himself the task of bringing order into this field by surveying the experimentally determined properties of thin films and relating them to physical theory. Herbert Mayer is Professor of Physics at the Mining Academy of Clausthal, where he conducts research into the properties of thin films devoting particular attention recently to the optical and electrical properties of alkali metal films.

Several years have elapsed between the publication of the two volumes reviewed here and possibly the division of the subjects treated would have been different had they been published simultaneously. Thus in Volume I is discussed the preparation of thin films, thickness measuring techniques and the optical properties of dielectric and metal films. Volume II deals with film condensation and crystalline structure, electrical conduction in, and the magnetic properties of, thin films. Your reviewer would have preferred to have seen the section on condensation and structure linked with the section on film preparation in Volume I, but this is probably pedantic and certainly is not intended to detract from the valuable treatment Professor Mayer has given to the subject. For example, Professor Mayer's treatment of thickness measuring techniques (reviewed up to 1950) is the most thorough available on the subject.

Volume II is the more ambitious work as is shown by the number of literature references which reach the thousand mark. Here we start by studying the nature of the forces between atoms and molecules absorbed by surfaces and their influence on the

conditions for film condensation, the film texture and crystalline state. Then follow chapters on the electrical resistivity of thin films, in which the influence of nearly every known agency is considered, *e.g.* nature and temperature of base, gas contamination of the support and film, and disorder in the crystal lattice. The discussion on the magnetic properties of thin films should be of interest not only to the research worker but also to the designer of electronic computers because of the recent use of thin magnetic films as storage elements.

With 716 pages of detailed matter to consider, one could not help but have some criticisms. One of these is that Professor Mayer is primarily interested in developing the theoretical aspects of the subject, and when dealing with the experimental properties of the films under review, he is inclined to give limited attention to the deposition technique used in their production.

The section on electrical conduction in thin metal films contains an extensive history of the experimental work carried out over the last fifty years. One learns that the resistivity of a very thin film may be exceptionally high, either because it possesses an uneven texture or because electrons lose energy by collision with the smooth boundaries of an ideal layer. Perhaps, however, Professor Mayer will be able in future editions to include a discussion of the recent work on gold films on oxide supports, which possess a more smooth texture than usual and have a resistivity tending to that of the bulk metal even when the layer is only 50 atoms thick. In fact, the small rise in resistivity can still be accounted for by atomic irregularities in the film boundaries and inelastic collisions of electrons with the boundaries need not be considered.

Most of the uses of thin films and research work into their properties employ glass as a film support and your reviewer would have liked Professor Mayer to devote more attention to the influence of this material on the mundane problems of film stability and durability. However, the discussion of the crystalline structure of thin films is thoroughly documented and should be useful to those interested in oriented over-growths.

A survey of the literature references quoted in the two volumes showed that out of a combined total of 1,600 only 30 referred to work in the Soviet Union and of these 25 were for pre-war papers. (This is not a criticism of Professor Mayer alone; your reviewer has been criticised for similar reasons.) However, from the present signs of the advanced state of science in Russia, it is impossible to believe that thin film technology is not well developed there. Professor Mayer has probably suffered from the same problems as workers here who rarely can read Russian, and who find it difficult to obtain translations and at times the original papers. It is even possible that the Russians published less in this field after the war, but they certainly are publishing now as examination of Physics Abstracts shows. Fortunately there are signs that English translations of Russian papers will be more readily available in the future.

Perhaps some of the answers to the problems raised here will be given in the third volume of Physik Dünner Schichten which Professor Mayer promises us in his introduction to Volume II. In the meantime, your reviewer (rather belatedly) proposes that these two volumes are indispensable to any laboratory engaged in making, using or studying thin films.

L. HOLLAND.

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The abstracts appearing in Vol. VII & VIII, dated January, 1959 are the accumulation of abstracts submitted during 1957-58.

VACUUM
CLASSIFIED ABSTRACTS

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Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

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All abstracts published in this section are allocated to one or more of the subjects shown in the following list

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Translations

Translations of some of the foreign publications quoted can be supplied.

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I — General Science and Engineering — I

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10 — VACUUM ENGINEERING (GENERAL) — 10

The Early History of the Vacuum Pump

1/I

United Kingdom. Galileo first pointed out that a pump could not raise water through more than about 1,060 cm. In 1644 his student, Torricelli, used the evacuated space above a barometer to carry out elementary experiments. The first machine which would produce a progressive vacuum was designed by Guericke. It consisted of a piston sliding in a cylinder having two valves, an inward opening one at the vacuum end and an outward opening one, half-way along its length. Several hours were needed to evacuate a receiver. A pump of this description was used by Guericke in his celebrated Magdeburg sphere experiment. In an improved form of the pump, a lead washer was used for sealing, and the wooden piston had string packing. The valve was of leather, closed by a spring. An alternative valve consisted of a peg, removed by hand, which was operated when the air pressure had become too low to open the spring valve. Boyle and Hooke constructed a pump operated by a rack and pinion which did not have to be immersed under water to provide air-tight joints as did all the others of this period. It had a 3 inch. i.d. brass cylinder enclosing a wooden piston with a leather disc nailed to it. An oil-water emulsion was poured in for smooth running and airtightness. A 25 l. spherical glass receiver was the largest, of the necessary thickness, that could be made. A sealed opening at the top facilitated the introduction of objects for experiments. In Boyle's second pump the receiver was a bell-jar cemented, by a beeswax-turpentine mixture, to a flat plate. In 1674, Papin and Huygens also constructed a pump fitted with a bell-jar over a flat plate. The piston, driven by rack and pinion was sealed by tow, soaked in water, and covered by a layer of oil. An improved version by Papin incorporated the first two-way tap. Senguerd built a pump in 1679 with an oblique barrel and two-way tap which connected alternately with the receiver and atmosphere. Large numbers of this pump were made. The next improvement was a two-cylinder pump, designed by Papin, with the two pistons linked by a cord so that the descent of each piston assisted the raising of the other. In 1709 Hauksbee described a pump he had built which also incorporated two cylinders, which were worked by rack and pinion. A model of this pump, which is still in working order, has given regularly a vacuum of less than 1 inch water pressure. Hauksbee records having obtained $\frac{1}{2}$ inch water pressure. After this pump there was little change in design until the twentieth century.

Article by E. N. da C. Andrade *Endeavour* **16**, Jan. 1957 29-35

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High Vacuum Pumps

2/I

United States. This is a brief review article which discusses some of the main types of vacuum pumps and their applications in typical industrial processes. The pumps mentioned include single-stage or compound rotary pumps, oil or mercury diffusion pumps, and mechanical booster pumps. Refrigerated traps may be used in conjunction with any of these to reduce residual pressures further or to prevent contamination by volatile materials. Diagrams are given to show the working principles of rotary and diffusion pumps, and some typical speed/pressure curves for these two types. Their operating characteristics are then explained, and some of their limitations pointed out, such as the need to protect rotary pumps from contamination. Finally, a table is given which indicates broadly the type of pump, or combination of pumps, suitable for use with a variety of equipment ranging from vacuum furnaces to particle accelerators.

Article by Z. Dobrowolski *Prod. Engng.* Mid-October 1957 J10-J12

High Vacuum Pumps

3/I

(Reference only)

Article by Z. Dobrowolski *Chem. Engng.* **63**, Sept. 1956 181-184

The Application of the Centripetal Effect in Air to the Design of a Pump

4/I

Israel. The device described makes use of a phenomenon in air which, predicted first by Reiner and subsequently demonstrated by Weissenberg, has been known to exist for some time in the flow of a very viscous liquid such as rubber solution. When sheared between a rotating and a fixed plate, positioned parallel to, and co-axial with, each other, the liquid is drawn inwards against the action of the centrifugal forces. The central component of the present device consists of a motor-driven rotating plate facing a stationary plate, the whole assembly representing the basic design elements of a centripetal air pump. The distance between the two plates can be varied from 0.75 mm. to 0.015 mm. Fine adjustments are provided for rotor and stator supports to ensure strict parallelism and springs secure the adjustments and prevent backlash when the gap is varied. The rotor assembly is particularly rigid to avoid a dangerous degree of vibration particularly in the longitudinal direction. The lowest transverse vibration speed was 50,000 vibrations per minute which is well above the 10,000 r.p.m. maximum speed of the rotor. A hole at the centre of the stationary plate was connected by means of a nipple to a tube fitted with a manometer or flowmeter. This instrument, at the maximum gap width and a rotor speed of 7,000 r.p.m., registered a suction of about 1 cm. of water. When the gap was reduced to about 0.02 mm., a pressure of more than half an atmosphere was recorded.

Letter by B. Popper & M. Reiner *Brit. J. Appl. Phys.* **7**, Dec. 1956 452-453

On the Factors Determining the Final Vacuum in Vacuum Installations Fitted with Oil Diffusion Pumps

5/I

See Abstract No.: 14/II

Abstract No. and References

I — General Science and Engineering — I

Contd.

6/I

Article by J. Rothstein *Rev. Sci. Instrum.* **29**, March 1958 243-244

Rate of Exhaust Through a Tube or Orifice

United States. A formula for the rate of exhaust through a tube or orifice given in *Dushman* "Scientific Foundations of Vacuum Technique", is inconvenient to apply except in two limiting cases. The differential equation from which his formula is derived is solved in another way giving a simple hyperbolic co-tangent formula easy to discuss or apply over the entire range. It is shown that the formula permits of ballistic use of a tube or orifice to measure pressure surges too rapid or too large to be monitored in the usual way. Several applications are suggested to electron tube manufacture and vacuum processing of various materials.

(*Author*)

7/I

Letter by W. Steckelmacher *Brit. J. Appl. Phys.* **8**, Dec. 1957 494

The Flow of Gas at Very Low Pressures

(Reference only)

8/I

Vacuum Flow of Gases Through Channels with Circular, Annular and Rectangular Cross Sections

United States. Vacuum flow of hydrogen, helium, air, carbon dioxide, and Freon-12 was measured in copper channels. These channels included a circular section with a 3.64 cm. radius, two annular sections formed by inserting cores of 1.905 cm. and 3.174 cm. radii into the circular section, respectively, and a rectangular section 0.324 cm. by 22.86 cm. In each section the flow was found to approach the molecular-flow limit with decreasing pressure, but the degree of approach depended on the cross section. When the molecular-flow equation was given in terms of the Barrett and Bosanquet geometric factor, K,

$$Q_M = K(4/3)\ (A^2/O)v(dp/dx) = KQ_A,$$

the degree of approach became poorer as K increased. The data approached the slip-flow limit with increasing pressure in all the channels. The experimental data were fitted over the entire range with empirical equations of the form

$$Q = Q_L + KQ_A\left[\frac{1 + C_1\,(X/\lambda)^{C_2}}{1 + C_1\,16K/3\pi\,(X/\lambda)^{C_2}}\right]$$

where Q_L is the laminar flow term, and X/λ the Knudsen number. The data from the rectangular section were fitted with $C_1 = 1.23$ and $C_2 = 0.3$. A general empirical equation for annular and circular sections was obtained when we set $C_1 = 0.275 + 1.27b/a$ and $C_2 = 0.4$. The fraction of molecules diffusely reflected from copper surfaces was found to decrease with increasing molecular weight. The absolute values of f could not be determined from the data. However, when $f = 1$ was assumed for hydrogen, the following values of f could be calculated by comparison for the other gases: Helium, 0.99; air and carbon dioxide, 0.97; and Freon-12, 0.90. Equations for slip flow in annular and rectangular sections were derived. Also derived was an approximate slip-flow equation for annular sections that agreed well with the rigorous derivation for channels with ratios of radii greater than 0.5. A study of the effect of the molecular nature of the gas on slip flow indicated that, with decreasing pressure, a positive deviation of flow from the slip-flow equation results. This would explain the apparent decrease in f observed in the slip-flow region. A simple approximation of the flow in the molecular-flow region was given that consisted of adding to the self-diffusion solution at low Knudsen numbers an additional flow due to streaming. This is expressed by

$$Q = (1 + 4A/\lambda\,O)\,Q_D$$

where A is the cross-sectional area of the flow channels, λ is mean free path, O is periphery of the flow channel, and Q_D is the flow rate due to diffusion or self diffusion.

(*Author*)

Report by W. Dong *A.E.C. Rep. No.* UCRL 3353 April 1956

10/I

Article by D. S. Stark *Brit. J. Appl. Phys.* **8**, May 1957 210-213

The Conductance of a Column of Vapour for Gases at Low Pressure

United Kingdom. Vacuum systems often contain stationary columns of vapour. A general expression is derived for the conductance of such a column for gases at low pressures. A rough experiment using columns of mercury vapour yielded values for the conductance for air and hydrogen which were of the same order of magnitude as the values predicted from the expressions given. It is shown that the conductance of the vapour can be less than that of the pipe which contains it. This becomes increasingly probable as the pipe diameter and the vapour pressure are increased.

(*Author*)

11/I

Ultra-High Vacuum Techniques

See Abstract No.: 119/II

12/I

Ultra-High Vacua

United Kingdom. This paper deals with the production and measurement of pressures below about 10^{-6} mm. Hg, at which pressure the limitations of diffusion pumps become apparent. The work of Nottingham, Alpert and others on the lower limit to the pressure range of a conventional hot-cathode ionisation gauge is discussed and improved gauges of the Bayard-Alpert type are described together with recent modifications due to Nottingham. In these the release of photoelectrons from the collector by x-rays generated by the electrons

I — General Science and Engineering — I

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Abstract No. and References

arriving at the accelerating electrode is reduced to a negligible amount. This extends the lower limit range of the gauge from about 10^{-8} to 10^{-11} mm. Hg. Next, isolation valves are described; these cannot use grease and they must be able to withstand a temperature of 450°C while the system is being outgassed. Most designs are modifications of the valve introduced by Alpert in which the seal consists of a conical Kovar nose-piece which is driven by a force of about 1 ton into an orifice in a copper block; the hard Kovar beds into the softer copper and produces a good vacuum seal. A simpler greaseless glass isolation valve using standard spherical ground glass joints is also described. The action of the ionisation gauge as a pump is considered and an estimate made of the pumping speed which should be obtainable. The theoretical value agrees well with those quoted by Alpert. The results of further experiments reported here indicate that Alpert's claims are justified provided that the initial pressure is sufficiently low. Suggestions are made regarding the disposition between Alpert's results and those of other workers. The omegatron is described and a short survey is given of those factors which set the limit to the lowest pressures attainable in a glass envelope.

Article by J. Yarwood *J. Sci. Instrum.* 34, Aug. 1957 297-304

Modern Developments in the Techniques of Vacuum Pressure Measurements 13/I

See Abstract No.: 37/II

On the Measurement of Very Low Gas and Vapour Pressures 14/I

Germany. Of the various instruments available for the measurement of lowp ressures, only the ionisation gauge in the form as modified by Alpert is supposed to give accurate readings in the pressure range below 10^{-8} mm.Hg. Recently an alternative method has been suggested and employed for the purpose which is based on the changes in the pattern of field emission from a freshly annealed tungsten point as a result of the progressive formation of a monomolecular layer of the gas, present in the vessel, on the tungsten point. The time taken to obtain a predetermined pattern of the field emission from the tungsten point is a measure of the pressure prevailing in the vessel. The authors, engaged in experiments involving the use of a tungsten point in a highly evacuated vessel, noticed that the readings obtained by the field emission method do not necessarily agree with those obtained with the ionisation gauge. This caused the investigation reported here. The vessel was a glass tube of 10 cm. diameter fitted at one end with a filament finishing in an etched point. Connected to the glass tube was an ionisation gauge and a mercury diffusion pump *via* an isolation valve. All determinations were effected with the isolation valve closed. Reference is made to investigations by Drechsler and Hess who obtained a predetermined emission pattern after a period of 300 sec. which corresponded to a pressure of 10^{-7}mm. Hg as established by other means. The authors required 10 hours to obtain the same pattern in their own experiments. Accordingly, the pressure should have been 8.5×10^{-10}mm. Hg. Parallel measurements with the ionisation gauge gave $8–9 \times 10^{-9}$mm. Hg. Thus there is a considerable discrepancy between the findings of Drechsler and Hess and the authors, and between the recordings by the ionisation gauge and the field emission method. Regarding the latter difference it should be taken into account that the ionisation gauge reading is a function of the product of the particle numbers per unit volume and the mean value of ionisation probability whereas determinations by the field emission method depend on the rate of deposition as a function of the product of the number of particles hitting the point and the adhesion probability. Assuming the adhesion probability of pure oxygen as 1 the authors calculated conditions for air at 10^{-7}mm. Hg and came to the conclusion that the deposition of the gas layer on the tungsten point should be completed after 100 sec. whereas Drechsler and Hess recorded a period of 300 sec. and the authors registered a time of 3,000 sec. The authors further observed that absorption after that interval was still far from complete indicating that the pressure recorded by the ionisation gauge may have been that of some gas other than air, a condition where equivalent readings by the two methods could no longer be expected. The following phenomenon appeared to confirm this. After closing the isolation valve the ionisation gauge first recorded a comparatively high pressure. This was followed by a reduction fairly rapid at first and slowing down in the end. These are the normal pressure recording characteristics of the gauge attributed to the pumping action of the gauge. However, parallel observation of the field emission pattern indicated an acceleration instead of a slowing down of the deposition rate. It seems that the residual atmosphere, the pressure of which is recorded by the ionisation gauge, consists of material at low volatility which is merely driven off to the cooler regions of the system instead of being 'pumped' away.

Article by F. Kirchner & H. Kirchner *Z. Angew. Phys.* 8, Oct. 1956 478-481

Barium Absorption Pumps for High-Vacuum Systems 15/I

United States. Describes a gettering system in which the action of evaporated barium is assisted by the use of a source of ionisation, which enables it to deal with the noble gases and with organic vapours. Advantages claimed for the barium pump are the elimination of diffusion pump vapours and cold traps, and also of the need for a continuously-running backing pump. The backing pump is used to initially reduce the pressure to about 10^{-2}mm. Hg, and the evaporation of the Ba will then lower it below 10^{-6}. An example is quoted of a high-voltage tube without gaskets, in which a pressure of 3×10^{-7} mm. Hg was maintained for one year. In a tube with 10 polyethylene and 2 rubber gaskets, the Ba had to be reactivated by a new evaporation every month. An example of a barium pump is sketched and described. It consists of a stainless steel tank 10 inch. long by $4\frac{1}{2}$ inch. diameter, with a 1,200 W heater wound round its lower (closed) end, and a water-cooling coil round the upper part. The condensation area is increased by the use of stainless fins. The tank can hold as much as 1 lb. of Ba, which can be evaporated at a rate of 1 oz. in about 10 min., the temperature being maintained within 25° of 800°C. The upper end of the tanks leads *via* a valving system to the tube to be pumped and to the backing line, while a Penning gauge is attached to the header to measure the pressure and provide

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Article by R. W. Cloud, L. Beckman and J. G. Trump *Rev. Sci. Instrum.* **28**, November 1957 889-892

a source of ionisation. The performance was measured by admitting metered quantities of various gases through a capillary leak, and observing the pressure for various rates of leakage. The pumping speed was found independent of pressure in the range 5×10^{-6} to 5×10^{-4} mm. decreasing exponentially with the total quantity absorbed. Speeds quoted range from 0.01 l./s. for Ar to 140 l./s. for pure H_2, with a half-value capacity in the range 20–600 mm.-l. An exponential equation relating speed to quantity absorbed is proposed, similar to that found for oxidation of metals in dry air.

16/I

Barium Getters and Carbon Monoxide

Article by R. N. Bloomer *Brit. J. Appl. Phys.* **8**, Sept. 1957 352-355

United Kingdom. The absorption of carbon monoxide by evaporated barium films has been studied in a pressure range 10^{-6}–10^{-7} mm. Hg. The mechanism is shown to be similar to that for oxygen (Bloomer) and is explained with the aid of Mott's theory. Carbon monoxide is dissociated at the surface of the barium film, and an oxide layer containing free carbon is formed. Below 80°C the layer of reaction products is protective once it has grown to a thickness of about 50Å. Above 80°C the whole of the barium is used up in the reaction. The condensation coefficient at room temperature is 0.012. Thus, about 1 in 80 of the carbon monoxide molecules striking the getter surface in any period is taken up by a barium film at room temperatures (1 in 40 at 170°C). The condensation coefficient is limited to these low values by the superficial density of interstitial barium ions in the free surface of the oxide layer. A nearby ionising discharge has very little influence upon the fraction of incident molecules which is condensed upon, and taken up by, a barium film.

(*Author*)

17/I

Reaction of Carbon Monoxide with Evaporated Barium Films

Letter by R. N. Bloomer *Nature* **180**, 3.8.1957 249-250

United Kingdom. The taking-up of carbon monoxide by evaporated barium films has been studied to discover whether there is a critical temperature above which the barium is not poisoned by the gas. The critical temperature above which the reaction product ceases to be protective has been found to be 80°C. Above this temperature barium getter films continue to react until used up completely. The reaction appears to be one of oxidation in the presence of carbon due to dissociation of the gas.

18/I

Absorption of Oxygen and Carbon Monoxide by Barium Alloy Getters

United Kingdom. Reference is made to experimental work reported by Wagener (see Vol. I, Abstracts No. 23/I and 91/I, and Vol. IV, Abstract No. 145/I) on the pumping speed and capacity of barium alloy getters of the type used in radio valves. The result of this work indicated, but did not finally establish, a relationship between pumping speed of the getter and the amount of ionising electron current passing through a diode or ionisation gauge situated nearby. The author reports on experiments carried out by himself to explore this problem further. The apparatus used was similar to Wagener's but was fitted in addition with a Pirani gauge to be employed for measurements when the ionisation gauge was switched off. The getters tested were of the KIC type, of 20 mg. size with a non-magnetic stirrup as normally employed in television tubes. A table, partly reproduced below, gives the results for pumping oxygen at varying electron currents. The table shows

Electron current (μA)	0	5	10	100	1,000
Speed of pumping (l./sec.)... ...	1.50	1.50	1.50	1.54	1.85

that barium getters pump oxygen at zero electron current. A graph is shown which indicates that the speed of pumping increases with the temperature. The capacity of the getter film (8 sq. cm. area in the present experiment) is independent of its thickness at room temperature but increases with the thickness at higher temperatures. The author found that the presence of a hot tungsten filament (above 1,800° K) near the getter counteracts the falling of the speed as the end of the life of the getter is approached. Switching off of the filament at this stage will cause pumping of the getter to cease immediately. It is suggested that atomic oxygen is formed at the hot tungsten filament which has a catalytic effect on the taking up of oxygen by the getter at that stage. Further experiments showed a considerable increase of pumping speed and capacity with temperature when carbon monoxide was used as the test gas. These results are at variance with those of Morrison and Zetterstrom (see Vol. V, Abstract No. 18/I).

Letter by R. N. Bloomer *Nature* **178**, 3.12.1956 1000-1001

19/I

The Oxidation of Evaporated Barium Films (Getters)

Article by R. N. Bloomer *Brit. J. Appl. Phys.* **8**, August 1957 321-329

United Kingdom. Mott's theory of the oxidation of metals is summarised and expressed in terms comparable with the throughput, capacity and speed of pumping, which are measured on the present work on barium getters. It has been found that there is a critical temperature, about 40°C for barium, below which a thin protective film of oxide is formed. Above 40°C barium films oxidise right through, and so in this case the capacity of barium for oxygen depends on the amount of metal used. The feature of the mechanism which limits the speed of pumping of oxygen has been found, and hence the speed has been increased in various ways. The theory of Mott concerns only the growth of an oxide film that is already at least a mono-layer thick. In the present study it has been found that the first mono-layer spreads from nucleation centres on the barium surface.

(*Author*)

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Abstract No. and References

Barium Getters and Oxygen

20/I

United Kingdom. The sorption of oxygen by films of barium evaporated in a vacuum has been studied, particularly with a view to determining the influence of nearly ionising electron current. Previous work by Wagener and other workers is reviewed and it is pointed out that some of the earlier investigations had led to contradicting results. The speed of pumping and the capacity of the films were measured using commercial-type getters which could be heated in a controllable fashion from a low-voltage mains transformer. The speed of pumping was determined by measuring the pressure difference across a capillary tube of known conductance by means of two ionisation gauges of the Bayard and Alpert type. Subsidiary experiments were carried out to compare the sensitivity of the two gauges and to show that the pumping action of the gauge adjacent to the getter was negligible under the conditions of the main experiment. The capacity of the getter was determined by multiplying the area under the pressure difference *versus* time curve by the conductance of the capillary tube. The capacity was converted into an equivalent number of mono-layers on the apparent surface of the film, on the assumption that the film was smooth down to atomic dimensions and that the gas molecules were packed together as closely as possible. It was found that the speed of pumping was almost independent of the ionising electron current in the oxygen gas. This confirms that the Wagener hypothesis (*Brit. J. Appl. Phys.*, **2**, 1951, 132) is untenable and shows that the take-up of oxygen by barium follows laws different from those for the cleaning-up of residual gases by low current electric discharges alone. However, when the speed of pumping begins to fall, oxygen is still taken up if some of it comes first into contact with a nearly incandescent tungsten filament. Measurements of the variation of speed in the early life of the films, and of the influence of evaporation conditions upon pumping, showed the importance of nucleation centres at which oxidation starts. The increase of speed and capacity with temperature show that these are limited by diffusion processes within the getter film.

Article by R. N. Bloomer *Brit. J. Appl. Phys.* **8**, January 1957 40-43

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Reaction Between Oxygen and Hot Gold

21/I

United Kingdom. The reaction between oxygen and hot gold was observed during a general investigation into the rate of disappearance of oxygen at a pressure of a few microns in the presence of hot metal filaments. Using a pure gold filament heated to 900°C and a clean Pyrex vessel, the rate of deposition of gold on the Pyrex walls was found to be twenty times that expected from the vapour pressure of the gold, and this rate is constant. The rate of disappearance of oxygen decreases asymptotically. The film deposited on the walls was found by x-ray diffraction to consist of pure gold. When the walls were warmed the oxygen disappearance rate increased by 50% between 20°C and 50°C. The rate also increased after an overnight 'rest'. Finally, the reaction rate increased with filament temperature. The authors suggest that the oxygen first forms a gold oxide on the filament surface; this evaporates and is deposited on the walls of the vessel but becomes unstable as the thickness of the deposit increases. Further gold oxide arriving at the walls breaks up to give a gold deposit plus free oxygen atoms. Some of these oxygen atoms are adsorbed on the surface or on impact with the walls. The increase in oxygen disappearance rate on resting may be due to slow migration of oxygen or oxide to more sheltered surfaces. The work is being continued.

Article by L. G. Carpenter and W. N. Mair *Nature* **179**, 26.1.1957 212-213

The Use of Getters in Magnetrons

22/I

Holland. Experiments were made in which the pressure in a magnetron was varied whilst it was working, and it appeared that changes within the range of 10^{-6} to 10^{-4} mm. Hg had little effect on output or stability. It was found, however, that a considerable improvement in shelf life could be obtained by using a getter. Instead of the usual 'seasoning' every 3 months, to take up gas released during storage, magnetrons which had been stored for as long as one year could be started at full voltage. It was also found that arcing, which spoils the cathode and damages the straps, was materially reduced, so that life-limiting factors other than cathode emission are of less importance. A further advantage is that the ageing time of new tubes is reduced and the frequency drift during ageing is more predictable, while the side-lobes in the frequency spectrum are improved. The type of getter used is not stated.

Article by P. Zijlstra *Le Vide* **12**, Sept.-Oct. 1956 248-250 11/I

Trapping of Helium Ions and Re-Emission of Trapped Atoms from Molybdenum

23/I

United States. The authors present further experimental results to those described in Abstract No. 14/I Vol. 5, and also put forward a phenomenological theory to explain the processes of trapping and re-emission of helium ions on molybdenum. The saturation phenomena previously noted and attributed to re-emission were investigated more fully and it was found that a reduction in the number of trapping sites did not explain the phenomena. The rate of re-emission was found to be proportional to the number of gas atoms trapped over a range corresponding to surface coverage of from 0.3 to 20% of a monolayer. Explanation of the results obtained rules out the existence of any simple physical trapping mechanism but rather suggests the penetration of ions into the surface with the subsequent re-emission controlled by a diffusion process to the surface. A pumping theory is developed starting from the expression

$$\frac{dn}{dt} = -\frac{n}{\tau} + r(t)$$

(where r(t) is the re-emission function) and on the basis of short and long term re-emission occurring. The short term re-emission at fractions of a second cannot be observed experimentally but comparison of the final expression for $\frac{dn}{dt}$ with experimental results suggests that the short and long term re-emission have essentially the same physical basis. The theory satisfies experimental results for accelerating potentials up to 2,100 V and for surface coverage approaching a mono-layer. In conclusion the authors state that the clean-up phenomena of helium in molybdenum can be explained by the basis of ionic pumping or trapping in which re-emission plays an important part.

Article by L. H. Varnerin & J. H. Carmichae *J. Appl. Phys.* **28**, August 1957 913-919

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24/I

'Clean-Up' of Radioactive Gases Used for Surface Studies

Czechoslovakia. Experiments are described in which radon was 'cleaned-up' into the surface of glass plates by an h.f. discharge produced by applying a Tesla coil to the glass wall of a small cell containing air at a pressure of 15-20 mm. Hg and a few microcuries of radon. The presence of the radon on the surface was then detected by taking an auto-radiograph. In the conditions of the experiment the radon had an effective half-life of 3 days at ordinary temperatures, or a few minutes at 310°C. It was found that nearly half the short-lived products of the α-decay of radon would transfer themselves to a second glass plate placed in contact with the first. Thus, considering the limitations of re-coil effects, the radon atoms must be within 200 Å of the surface of the glass. Grinding the surface of the glass considerably reduced the clean-up, while even a single mono-layer of calcium stearate gave a detectable reduction. The effect can thus be used in the study of mono-molecular films and may be useful for evaporated metal films as well. The method can also be used in the preparation of samples of radioactive gases for measuring their soft radiation or investigating alpha-recoil.

Letter by C. Jech *Nature* **178**, 15.12.1956 1343-1344

25/I

High-Sensitivity Mass Spectrometer for Noble Gas Analysis

See Abstract No.: 82/II

26/1

Physics Meeting

Germany. Summaries of the following papers presented in the section Vacuum Physics at the Physics Meeting lasting from 27.9 to 1.10. 1957 in Heidelberg are given: *K. Ziock:* Measurement of back diffusion of oil in oil diffusion pumps. *K. Brill:* On the discharge process of rotary vacuum pumps. *G. Reich* and *H. G. Nöller:* Analysis of final pressure of oil diffusion pumps with an omegatron. *K. G. Günther:* On the intensity distribution of vapour and molecular rays. *K. G. Günther:* On the condensation behaviour of high-boiling substances.

Report by Anon. *Chem. Ing. Tech.* **30**, 1958 262

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27/I

Equilibrium Between Glass and Water Vapour at Bake-Out Temperatures

United States. Reference is made to experiments on the diffusion of water through glass under vacuum reported previously (see Vol. VI, Abstract No. 79/II). The present article reports further experiments carried out on the same problem and conducted with soda-lime glass (Corning 0080). The samples, having a uniform concentration of water throughout the glass at the outset of the experiments, were pre-heated in atmospheric air containing water vapour at various partial pressures ranging from 0.5 μ to 63 mm. Hg. The samples were then rapidly cooled, transferred to the vacuum system and heated up again to 500°C. Readings during the vacuum bake-out were taken after the first half hour, a further 3 hours and a total of 6½ hours. Preheating in atmospheric air was carried out either at 500°C for 2 hours or 550°C for 1 hour. Glass rapidly cooled in atmospheric air is known to adsorb 2.5 μ l./dm.² of water. This amount therefore has been deducted from the readings recorded during vacuum baking. The observations are shown in graphs where the volume of the evolved water is plotted against the square root of the baking time. The performance of a non-preheated sample during the bake-out treatment served as an indication of whether the various preheated samples had taken up or diffused out water during the preheating treatment. By plotting the differences only of the amounts of water evolved or given up between preheated samples and non-preheated samples during the 6½ hour vacuum bake-out, against the partial pressure of the water vapour in the atmosphere during preheating, and showing a gain of water by a positive value and a loss of water by a negative value, straight lines are obtained. At the intersections of these lines with the horizontal zero water-gain/loss line the pressure value shown on the abscissa is the corresponding equilibrium partial pressure of the water. If the partial pressure of the water in the surrounding space is greater than this value water diffuses into the glass; if it is smaller, water will diffuse out of the glass. The rate of diffusion depends on the magnitude of the difference and on the temperature. The process appears to be completely reversible. It has further been observed that if the glass is heated in atmospheric air which is completely dry, the diffusion phenomena are the same as those observed in a vacuum bake-out. A combination of preheating in dry atmospheric air with a short heat treatment in vacuum might prove useful in processing valves where high envelope temperatures are to be employed.

Article by B. J. Todd *J. Appl. Phys.* **27**, Oct. 1956 1209-1210

28/I

A Simple Gas Inlet for Use With Vacuum Systems

(Reference only)

Note by J. Almond *J. Sci. Instrum.* **35**, Feb. 1958 70

29/I

Constant-Pressure Leak-Rate Gauge

United States. A method of measuring the rate, at which a gas is admitted to a vacuum system has been developed. The method allows the measurement of gases other than air, at different reservoir pressures. The amount of gas admitted is determined by the rate at which a mercury plug moves through a horizontal glass tube calibrated in terms of volume. The instrument has been useful in determining ion gauge sensitivities and diffusion pump speeds for various gases.

Note by K. W. Ehlers *Rev. Sci. Instrum.* **29**, Jan. 1958 72

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Improvements In the Use of the Porcelain Rod Gas Leak — 30/I
See Abstract No.: 126/II

Leak Detector for Hermetic Seals — 31/I
See Abstract No.: 157/II

New Ultra-High Vacuum Valve — 32/I

Czechoslovakia. A valve has been made using molten indium as a cut-off. Indium has been chosen on account of its good adhesion to glass, its low melting point and its low vapour pressure. To open and shut the valve, the indium must be heated to above its melting point, then allowed to cool slowly. The resulting seal to the glass walls will withstand atmospheric pressures, and at ultra-high vacuum, a pressure as low as 10^{-11} mm. Hg may be reached.

Note by L. Paty & P. Schurer *Rev. Sci. Instrum.* 28, August 1957 654-655

Counterbalanced Bellows in Vacuum Systems — 33/I

United States. Bellows are frequently employed as connecting elements between the metallic vacuum system and the line leading to the vacuum pump in order to allow for limited expansion and the movement required for alignment. Also, the bellows serve to reduce transmission of the vibration originating in the pump. If inserted directly into the line however, the bellows may collapse under the effect of the external pressure but, if made rigid, it will no longer function as desired. The authors describe briefly (and show in an illustration) an arrangement which overcomes this difficulty. A T-piece is inserted in the pumping line fitted with the bellows. One end of the 'T' is joined to the bellows and a second bellows is carried at the opposite end. The remote ends of the two bellows are rigidly linked. A connection of this kind will float freely while any stress or strain on the bellows in the pumping line is automatically counterbalanced by the action of the second bellows.

Note by C. B. Leffert, W. H. Kasner & T. M. Donahue *Rev. Sci. Instrum.* 27, Dec. 1956 1084

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Improvements r.t. Vacuum Hose Couplings — 34/I
See Abstract No.: 155/II

Improvements in Vacuum Control Systems — 35/I
See Abstract No.: 154/II

German Standards, Vacuum Technique. Concepts and Definitions — 36/I

Germany. Proposals for DIN 28400.

Report by Anon. *Chem. Ing. Tech.* 29, 1957 358-359

High Energy Nuclear Physics — 37/I
See Abstract No.: 107/I

High Vacuum Engineering — 38/I

United Kingdom. The role of high vacuum in the development and application of atomic energy is presented.

Article by F. D. Edwards *Financial Times* 1.7.1957 93

Vacuum Techniques in the Atomic Energy Industry I. — 39/I

United Kingdom. Improved vacuum techniques which have accompanied the development of U.K. atomic energy projects, such as the Capenhurst diffusion plant, the Dounreay fast reactor and the Calder heat exchangers, are outlined. In particular vacuum joints and valves, methods of vacuum testing and leak detection and a statistical quality control method suitable for the components of vacuum plant are described. Vacuum plant should be constructed, whenever possible, from rolled plate or forgings. Castings are not satisfactory as vacuum envelopes although under certain conditions castings are acceptable inside vacuum systems. Glass can hold large quantities of water on the surface and in solution, but owing to its high equilibrium vapour pressure, 10 mm. at 500°C, prolonged heating of glass in dry air should be adequate to remove most of the moisture. Vacuum connections should preferably be made by welded joints rather than flanges. An improved coupling illustrated comprises two identical flanges which are brazed to the vacuum pipe in a jig by induction

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heating. Another type of joint features two concentric sealing rings in the flange. A drilling in the flange connects with the space between the two rings and by a suitable pipe connection this 'interspace' between the rings can be evacuated. The joint is vacuum-tested by evacuating the small volume formed by the interspace and determining its pressure rise on isolation. It is not necessary to evacuate the whole plant. Flanged joints with a hexagonal copper sealing ring have remained vacuum tight up to 200°C after extensive temperature cycling, and a fully annealed nickel sealing ring, ⅛-inch square, has proved satisfactory for use in a flange joint in pipe of 2¼ inch. dia. carrying water at 2,500 p.s.i. and 350°C. Fully trapped indium metal O-rings are suitable for low temperature work. A bellows-type vacuum seal widely used in atomic energy work consists of a stack of thin stainless steel 'washers' joined by circumferential welds. In certain high pressure-high temperature work valve spindles have been sealed with ordinary steam packing, but a second seat, back seat, has been introduced against which the valve plug seats when fully opened. The packing is thus exposed to the working fluid only during operation of the valve. In vacuum testing and leak detection, the isolation pressure rise method gives a direct but slow indication of a leak. Mass spectrometers used in conjunction with a probing gas provide sensitive leak indicators but are too expensive and difficult to service for general use. The differential Pirani gauge method has been found suitable for laboratory work but in plant use trouble is caused by the absorption of impurities on the chemical absorbent interposed between one gauge and the vacuum system. Similar poisoning may occur in the hydrogen palladium gauge. A leak-detecting method which has proved reliable and simple is to place a charcoal trap, cooled with liquid nitrogen or solid carbon dioxide, between a Pirani gauge and the vacuum system under test. This trap ensures that the gauge preferentially responds to hydrogen. A calibrated standard leak for use with qualitative leak detectors is provided by the powder leak in which specially treated aluminium powder is packed between sintered metal discs and compressed by means of a bolt. The value of the leak is determined by the quantity and grade of the powder and the amount of compression. A statistical quality control method which has been used to 'pass' deliveries of vacuum equipment and plant components uses charts based on the binomial distribution. Each chart shows the limits of variation due to chance which may be expected in the number of 'passes' found in any random sample of given size. Separate charts are prepared for 50, 60, 75 and 90 per cent. pass rates.

Paper by O. H. Kronberger *Engineer* 15.11.57 702-705

40/I

Atomic Age Metals . . . Seeking New Outlets

United States. The article reviews the place in atomic energy, and the commercial prospects, of the new structural materials such as uranium, hafnium, beryllium and columbium (niobium) by considering the properties and uses of these materials in conjunction with the cost of the poundage produced. It is stated that zirconium having a combination of neutron transparency, corrosion resistance and high temperature strength is an ideal material for structural uses in reactor cores or for the cladding of uranium fuel elements. The development of zirconium is also briefly described together with its corrosion resistant properties against most acids and alkalies; its use as a rayon spinner being quoted as an example in this respect. The author expects the commercial nuclear market to be the outlet for hafnium which has a very high neutron cross section making it a good material for shielding and control rods. In addition it possesses corrosion-resistant properties comparable with those of zirconium besides having good mechanical properties and a high thermal conductivity. The high cost of beryllium is expected to cause its sparing use in commercial reactors although it can be used as a moderator, reflector or neutron source. Its listed properties include a specific gravity of 1.85, relatively high melting point, high strength at elevated temperatures, low neutron cross section and a high stiffness-weight ratio. One of the non-nuclear uses suggested for this material is the manufacture of aircraft structures although the limit of its structural value is the low ductillity of present-day products. The well known stabiliser in stainless steel, columbium, is stated to offer superior heat and corrosion resistance and to be suitable for the cladding of fast reactor cores and for the alloying of uranium to make fuel elements. The article closes with a resumé of the metals used in the nuclear industry as fuels, coolants and reducing agents.

Article by C. H. Chilton *Chem. Engng.* **64**, Sept. 1957 208, 210

41/I

A High-Vacuum Casting Furnace for Plutonium

See Abstract No.: 217/I

42/I

Vacuum Deposition of Metals

Article by Anon. *Nature* **180**, 20.7.1957 124

United Kingdom. Report of a meeting organised by the Department of Physics of the University of Cambridge and the British Scientific Instrument Research Association held at the Cavendish Laboratory on May 17th and 18th 1957.

43/I

Vacuum Engineering (and its Applications to the Coating of Surfaces)

France. The importance of vacuum technique in modern industrial and general economic life is discussed. Vacuum techniques are employed in (*a*) electrical engineering, (*b*) electronics, (*c*) nuclear research, (*d*) optics, (*e*) acoustics, (*f*) metallurgy and (*g*) biochemistry. Typical applications are in (*a*) the impregnation of cables and the drying of insulating materials; in (*b*) the production of thermionic valves (the world production of radio and television tubes is estimated at over 100 million); in (*c*) the operation of particle accelerators; in (*d*) coating of lenses by evaporation and sputtering techniques; in (*e*) certain aspects of the production of hi-fi equipment; in (*f*) the processing of refractory metals and the production of certain alloys, and in (*g*) the various techniques employed for the preservation of biological materials such as antibiotics, blood, etc. Vacuum techniques were employed in industrial production for the first time in 1881 by Paszburg in

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connection with the drying of sugar but the first to produce an artificial vacuum deliberately was Otto Guericke who conducted his famous experiment of the Magdebourg sphere publicly in 1657. The real basis for a successful industrial application of vacuum engineering was laid between 1905 and 1916 when Gaede developed the rotary pump, the molecular pump and the diffusion pump. Subsequent exploration of the uses of vacuum equipment and its progressive introduction in industry, particularly during the period 1920–1930, was largely influenced by Hickman's work. In principle, sub-atmospheric pressure should be measured in terms of the C.G.S. unit, the barye, which represents the force of 1 dyne applied to 1 sq. cm. area, but in practice pressure is measured in terms of height of the mercury column in mm., this unit being the basis of barometer readings and the barometer being the first pressure reading instrument in low pressure technique. The author proceeds to discuss the basic conception of the kinetic theory of gases. An atom has a size of the order of 10^{-8} cm. (1 Å). Solid materials contain approximately 10^{23} atoms per c.c. whereas materials in gaseous form contain about 2.7×10^{19} molecules per c.c. at atmospheric pressure and 0°C. Thus in a gas the interatomic distances are much larger and the forces effective between the atoms are less important. As a result the particles are in motion at a speed which is governed by the temperature and the mass. Collision of the gas particles with the wall of the container determine the gas pressure. The motion of the gas molecules which represents the kinetic energy of the gas is expressed by the formula

$$v = \sqrt{\frac{8\,RT}{\pi M}}$$

where R is the gas constant, T the absolute temperature and M the molecular mass of the gas. This demonstrates that the root mean square speed of a molecule is proportional to the square root of the absolute temperature also molecules of different types under identical temperature conditions have mean speeds which are inversely proportional to the square root of their specific mass. The incidence of collisions between gas particles themselves depends on the length of the mean free path which in turn is a function of the pressure. At atmospheric pressure the mean free path is 6.6×10^{-6} cm., at 10^{-2}mm. Hg the mean free path is 5×10^{-1}cm., and at 10^{-5}mm. Hg it is 500 cm. The product of the mean free path and the pressure of the gas is constant. Consequently if one is known the other can be calculated. (This article introduces a series of articles devoted to vacuum engineering applications such as coating.)

Article by R. Doré *Galvano* **229**, 1956 35-39

Vacuum Metallising

44/I

United Kingdom. The development of vacuum metallising is reviewed from the period before the last war when it was used in a small way to make optical reflectors, up to the present when it is replacing costly and tedious electroplating methods for some purposes, and opening the door to other applications hitherto considered impossible, impracticable or uneconomic.

Article by D. J. T. Kennedy *Industr. Finish.* 9, May 1957 554-559

The Uses of High Vacuum Evaporation for the Metallising of Textiles

45/I

Germany. In the introduction, reference is made to the Milium Process and to the modifications of the same developed by Farbwerke Hoechst AG. Essentially, the article deals with the deposition by evaporation on finished fabrics (as distinct from single threads). A Heraeus Plant suitable for the purpose is described in great detail. The width of the endless strip which can be coated is 400 mm. The authors then discuss certain physical properties of the coated fabric. Permeability of air is unimpaired in the case of evaporated coating, whereas in the Milium type of process it is reduced to one-third of the natural fabric. Glossiness of the coated material depends on the surface properties of the fabric. The textiles to be coated may be processed in the normal way prior to the coating—such as, for instance, trubenising. Adhesion on synthetic fabrics such as terylene, orlon, nylon, perlon, etc., is better than on cellulose fibres, and, in the case of the latter, adhesion on acetate silk is better than on copper rayon. Some details regarding adhesion tests are given. Absolute adhesion values depend largely on the residual water content of the dried fibre. A correlation has been found between the sorption value of the material, and the adhesion properties. If a metallised viscose fibre is treated with water, the metal layer will flake off after swelling. That is not so in the case of perlon. Abrasion resistance and non-shrinkage in washing the coated material can be improved by lacquering the material before and after coating. Pattern coating is possible. The optical properties of coated material have been investigated in the knowledge that light and, in particular ultra-violet light, produces a photo-chemical decomposition of the fibre material in the non-coated state. Coated materials show a large light absorption of the metal layer, preventing the light reaching the fabrics. This effect applies to both the Milium type of processes and high vacuum processes. The article concludes with a discussion of the heat conservation properties of coated fabrics.

Article by E. Elod & H. Jorder *Melliand Textilber. Heidelberg* 37, 1956 422-428

Evolution of Gas and Permeability to Gas of Constructional Metals for Vacuum Systems

46/I

Germany. Construction materials to be used in vacuum plant must meet the following two major requirements: (*a*) They must contain a minimum of gas either chemically bound, adsorbed, dissolved or occluded and (*b*) they must be absolutely gas-tight, have no pores and show no permeability. The most important single factor governing the suitability of the material is its surface properties. Glass is the most suitable material for the purpose but is gradually being replaced by metals. An important consideration is the vapour pressure of the metals at elevated temperatures. The saturation pressure of an evaporating substance as a function of temperature can be calculated sufficiently accurately for practical purposes from the equation

$$\log P_{mm} = 8.88 - 6\left[\frac{T_s}{T} + a\,\frac{T_s - T}{T}\right]$$

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where T_S (in °K) is the boiling temperature at atmospheric pressure, T (in °K) the experimental temperature and a is given by

$$a = \frac{S_x}{S_n} - 1$$

relating the entropy at boiling point of a normal substance S_n with that of the experimental substance Sx. The comparison substance may be represented by

$$\log P_{mm} = 8.88 - \frac{6T_s}{T}$$

Experimental observations evaluated by this method indicate that most substances have a vapour pressure of 10^{-3}mm. Hg at half their boiling point temperature. The partial pressure at which gaseous products resulting from dissociation or transformation may be present is related to the saturation pressure but the temperature at which the prospective equilibrium would be established is affected by other reactions which may have occurred at the same time within the envelope. Adsorption of gas by surfaces is governed by a variety of kinetic and energetic conditions which are not fully explored yet. The adsorption isobar characteristics of various metals are complex and indicate that an increase or decrease in temperature alone cannot lead to a gas free surface. At elevated temperatures reversible physical adsorption is replaced by irreversible chemical adsorption, the gas being retained in an atomic state in the metal. Chemical adsorption is the first step to permeation and dissolution in the metal. Chemical adsorption and actual combination cannot be readily distinguished. A mono-molecular adsorption layer may contain up to 10^{16} gas particles per square centimetre without being visible on the metal surface. Such a layer may contain about 10^{-8} mol. or 10^{-5} c.c. of gas per square centimetre surface area at N.T.P. Corrosion when first observed may have long passed the chemical adsorption phase. The adsorption of gas on surfaces in vacuum systems cannot be altogether avoided but can be minimised by removing substances such as moisture which promote chemical combination.

Getters and their characteristic properties

Getter	*Melting point °C*	*Evaporating temperature °C*	*Kind of gas*	*Gas-absorption c.c./mg. getter*[1]
Aluminium	658	1,400	Oxygen ...	38.6
Barium[2]	850	700	Oxygen ...	45
			Hydrogen ...	73
			Nitrogen ...	36
			Carbon Dioxide	60
Cerium-Lanthanum... (mixed getter to U.S. Pat. 1,720,005)	Ce 775 La 920	Ce 620[3] La 920[3]	Oxygen ...	51
			Hydrogen ...	64
			Nitrogen ...	16
			Carbon Dioxide	45
Magnesium	650		Oxygen ...	200
Thorium	1,850	500	Oxygen ...	33
			Hydrogen ...	54
Uranium	1,690	1,900[3]	Oxygen ...	9
			Hydrogen ...	21
Iron	1,530	1,600		
Potassium	63	240		

At the evaporation temperature in column 3, 1×10^{-3}g. of getter evaporates per sec. per cm.[2] of getter area. It is calculated by Langmuir from $T = MP^2 \times \frac{0.0034}{10^{-6}}$ where T is in °K, M = molecular weight and P is in mm.

[1] Maximum gas absorption with 'diffuser getter' which will be formed with 3 mm. rare gas pressure. After removal of this charge of gas the gas to be gettered was introduced at room temperature. The numbers represent the absorbed gas from 1 mg. of getter. (Measurements are from Ehrke and Slak *J. Appl. Phys.* 11, 1940, 129).

[2] The gettering of hydrogen by barium is reduced excessively by Hg vapour.

[3] P in the above equation was estimated from the formula $\log P = 7.9 - \frac{5Ts}{T}$

Adsorption phenomena of the type discussed are not to be confused with absorption phenomena deliberately promoted within an evacuated envelope to produce 'gettering' or 'clean-up' action. The latter processes usually involve the formation of a chemical compound. Little accurate data is available on the solubility equilibria of gases in metals. Solution of gases in metals is usually not a grain boundary effect but a homogeneous solution of the gases in the crystal lattice itself. As the lattice structure changes, *e.g.* in alloy formation, solubility values have also been observed to change. Hydrogen is almost invariably a major constituent of the gas content of metal. The causes of the presence of gases in metals are discussed. The time

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required for degassing a metal sheet to about 5% of its original gas content can be calculated from

$$Z_{Extr.} = \frac{2 \times 10^{-2} \times d^2}{3.6 \times 10^3 \times D} = \frac{d^2}{7.2 \times 10^5 \times D}$$

where D is the diffusion coefficient in cm.²/sec., d the thickness in mm. and $Z_{Extr.}$ the time in hours. The above formula applies only to the case of genuine solution. Permeability of a material is defined as the quantity of gas, in c.c. N.T.P., passing through one sq. cm. of surface per second at 1 atm. pressure difference and 1 mm. thickness of material. It can be expressed by

$$M_D = A\,(\sqrt{p_1} - \sqrt{p_2})\,e^{-E/T}$$

where p_1 and p_2 are the gas pressures on both sides of the metal wall and A and E are constants. In the case of metals $A = 1$. The author concludes with a brief discussion of capillary permeability for gas.

Article by E. Waldschmidt *Metall* No. 19/20, Oct. 1954 Translated by J. Standring *A.E.A. Rep.* No. IGRL-T/C 25 October 1956

47/I

Problems of Gases in Metals

United States. An understanding of the behaviour of gases in metals depends largely on having reliable methods for the determination of the kinds and amounts of gases present in the metal. One of the most widely applicable methods of gas analysis is the vacuum fusion method. It can be employed for the determination of oxygen and hydrogen and in some cases nitrogen. Details of the method are given. It facilitates a precision of 1 part per 1,000,000 of oxygen or 1 part per 10,000,000 of hydrogen by weight in a 10 g. metal sample. An alternative chemical method, less fool-proof than the vacuum fusion method, is the bromination method originally developed for use with titanium and zirconium but recently adapted for use with chromium, vanadium and steels as well. A further method for the determination of hydrogen in titanium and zirconium is the hot extraction method which permits evolution of hydrogen from the solid metal at 1,200°–1,450°C. The accuracy of the vacuum fusion method for the determination of nitrogen in metals is somewhat uncertain in cases where nitrides are likely to be formed. This applies in particular to metals such as titanium and zirconium. The most accurate results for nitrogen are obtained by a chemical procedure such as the solution-distillation method, in particular the micro-Kjeldahl technique. The most important gas metal problems in ferrous metallurgy are: (*a*) Embrittlement and porous weldments, (*b*) embrittlement of large castings, (*c*) blistering of coatings of porcelain, tin plate and electro-deposited metals, (*d*) reduced ductility and machinability of special steels. Porosity in ferrous weldments is known to be due to the carbon-oxygen reaction which cannot be properly controlled yet. Unkilled (not deoxidised) steel shows high temperature brittleness which can be overcome by deoxidation. However, too strong a deoxidation may cause reduced machinability. The presence of nitrogen in steel, while causing brittleness, is known to increase its strength at the same time. Case hardening by nitriding is beneficial. A high hydrogen content may produce (*a*) porosity during solidification, (*b*) flakes and shatter cracks, (*c*) reduced ductility and (*d*) delayed brittle fractures in high strength steels. In non-ferrous metallurgy the major gas metal problem is embrittlement. An addition of 1 part per million of oxygen to molybdenum already containing 2 parts per million of oxygen may destroy its ductility. Oxygen-induced embrittlement may occur in other metals with a low solid solubility for oxygen, whereas titanium and zirconium, for example, with comparatively high solubilities may be strengthened by the addition of small amounts of oxygen (a few hundredths of 1%) and will only become brittle if oxygen addition is excessive. On the whole, similar conditions apply in the case of nitrogen, but nitrogen content is more easily controlled than oxygen content which is governed by the presence of moisture. As in ferrous metallurgy, hydrogen content is the most important. In the case of titanium, existing specifications call for a maximum content of 150 parts per million in sheets and 125 parts per million in forgings. These specifications are being met in only about 90% of the titanium supplied today.

Article by M. W. Mallett *Mach. Lloyd Overseas Ed.* 10.11.1956 79-84 & *Steel* 139, 8.10.1956 95-98

48/I

Practical Processes for the Degassing of Light Alloys

France. The relative advantages of the degassing of aluminium alloys by (*a*) natural means, (*b*) bubbling with nitrogen and (*c*) bubbling with hexachloroethane are discussed. The natural evolution of gases, though time consuming, has the advantage of simplicity. The Gazal process of bubbling with nitrogen is less reliable and is best used in conjunction with natural degassing. Nitrogen degassing should be carried out in a furnace; formation of dross and inclusions can be reduced by subsequent treatment with manganese, titanium and boron. Reductions from 3.8 to 2.2% and from 1.8 to 1.3% are quoted. Bubbling with hexachloroethane has similar disadvantages to bubbling with nitrogen, but seems more reliable. Comparisons of cost are made for the case of degassing a charge of 50 kg. of high gas content, maintained at 800°C, to 75%, showing that natural degassing may cost between 50 and 140 francs depending on the furnace used, bubbling with nitrogen 96 francs and bubbling with hexachloroethane 84 francs.

Article by C. Mascré & A. Lefebvre *Fonderie* December 1956 496-508

49/I

Techniques in Metallurgy

Germany. The application of vacuum techniques in metallurgy results in the production of pore-free dense castings, high purity of the material, low content of dissolved gases, full efficacy of the alloying components due to the prevention of the formation of gas-metal compounds, and high accuracy in composition. Results of many authors of work on alloy and unalloyed steels are tabulated; in many instances results obtained by vacuum-melting are compared with those from normal melting methods. Many examples show considerable improvements in notched-bar impact strengths and lower transition temperatures. The effect of the vacuum on the content of dissolved gases in Fe and Fe-Cr is shown graphically. Improvements in coercivity and maximum permeability of Alnico 5 by the application of vacuum metallurgy and vacuum sintering of hard metals, ceramics and refractory metals (Ta, W and Mo) are mentioned briefly. The great improvement in the economy of these methods is shown by a lowering of treatment costs from 1 DM/kg to 0.1 DM/kg (from 1/8d to 2d.) in the last few years, mainly due to the employment of vacuum furnaces of capacity up to 20 t.

Article by W. Scheibe *Z. Metallkde.* 48, March 1957 91-100

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A Metallurgist's View of Metallurgy

United States. The scope of the work undertaken by the metallurgy department of the Bell Telephone Laboratories is illustrated by an outline of specific research projects and the practical developments which have resulted from them. One problem has been the development of an alloy suitable for springs but with an economical tin or nickel content. Statistical methods have been adopted for predicting the service behaviour of springs from a limited number of test data. As a result of work on plastic flow, laboratory equipment has been constructed which permits the extrusion of aluminium cable sheath over paper insulated cores, at temperatures that do not scorch the paper during recharging, and at pressures only about 10% higher than those used for lead in conventional equipment. The feasibility of extruding aluminium sheath commercially has thus been demonstrated. Results from basic research on plastic flow indicate that two specimens of the same metal deformed in different ways to a state of the same yield stress appear to exhibit the same rate of work hardening. The relation between plasticity and atomic scale defects such as dislocations, vacant atom sites, interstitial atoms and impurity atoms is being examined in three ways: By testing the behaviour of crystals exposed to ultrasonic waves; by investigating certain properties peculiar to germanium and silicon; and by a study of metal whiskers. Germanium and silicon crystals have the property that positions of dislocation can be revealed in the form of etch pits on specially prepared surfaces. In addition the presence of both vacancies and interstitial atoms can be detected by their effects on electrical conductivity. Semi-conductors such as silicon and germanium are used as crystal detectors in radar and microwave relays. More recent applications are the transistor, the silicon solar battery and the solid-state diode. The Bell Laboratories have prepared the purest known silicon and germanium using zone melting techniques. Their work on the preparation and study of pure single crystals is now likely to extend to the rare metals such as scandium, yttrium and the rare earths. Among the magnetic materials being studied are iron, manganese, bismuthide and barium-iron oxides (Ferroxdur). The development of ferrite core materials for inductors in communication equipment has reduced eddy current losses and made it possible to use higher carrier frequencies. Ferrites are also useful as isolators in microwave transmission systems. They can be used in one-way valve devices which permit a signal to pass towards the antenna, but prevent reflected waves from affecting the oscillator. Another possible use is in materials designed to have essentially square hysteresis loops. The magnetisation of these materials remains constant in direction and strength until a sufficient reverse field is applied. Then a sudden complete reversal of magnetisation occurs. Devices based on this property are of potential value for computers, magnetic storage and switching applications.

Article by E. E. Schumacher *Metallurgia* Sept. 1957 129-132

51/I

Method and Apparatus for Melting Metal under Vacuum

See Abstract No.: 127/III

52/I

The Application of Vacuum in Metallurgy

France. In melting and casting steel and iron the basic chemical phenomenon is the equilibration reaction between the iron and carbon

$$FeO + C \rightleftarrows Fe + CO$$

which governs the decarburisation and oxygen reduction alike. According to the law of mass action the equilibrium constant is

$$K = \frac{[FeO] \times [C]}{[Fe] \times [CO]}$$

As the iron mass in the melt is not perceptibly affected by the reaction and as the carbon monoxide partial pressure in the furnace atmosphere is constant it can be stated

$$K = [FeO] \times [C] \text{ or } K' = [O] \times [C]$$

i.e., the product of oxygen and carbon content in the melt is constant at any given temperature and pressure However as carbon monoxide is a highly volatile material a reduction of the pressure in the chamber results in a change of the value of the constant K′. For the case where K′ is 0.0025 at 1,600°C and 1 atm. pressure, and assuming a carbon content of 0.25% in the melt, the oxygen content varies with the pressure as follows: At 1 atm. 0.010%; at $\frac{1}{10}$ atm. 0.001%; at 1/76 atm. = 10 mm. Hg 0.00013%. Theroetically it is possible to reduce the oxygen content to nil by reducing the operational pressure to the required value, but the melt being in contact with the furnace walls silicon dioxide, if present in the lining, will dissociate independently of the prevailing pressure in accordance with the respective specific equilibrium constant thus counteracting the elimination of the oxygen by the main reaction. Similarly magnesium oxides, if present in the furnace lining, will dissociate, but the latter reaction is pressure-dependent and may become considerable at very low pressures. Even more important than the reduction of the oxygen content is the degree of decarburisation obtainable at medium pressures. Reverting to the previously quoted case of an oxygen content of 0.010% and a carbon content of 0.25% at 1 atm. the following conditions apply: At $\frac{1}{10}$ atm.—carbon content 0.025, at 1/76 atm. = 10 mm. Hg—carbon content 0.003%. In fact operation at reduced pressure facilitates the reduction of the carbon content in the presence of chromium, not obtainable when operating at atmospheric pressure. Thus, when operating *in vacuo*, chemical action alone is responsible for the manufacture of stainless steel with a carbon content of less than 0.02%. Recent development work has concentrated on exploiting to the full the physical action of vacuum. Vacuum melting on a large scale is somewhat restricted by the maximum size of vacuum furnaces so far available (1 to 2 tons). But Bochumer Verein have developed a method of vacuum casting (into ingots) of steel facilitating charges of 150 tons at a time (see Vol. VI, Abstract

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No. 45/I). The steel is melted in atmosphere and poured into a bucket fitted with a bottom pouring device. The bucket is placed above, and joined to, an evacuated chamber. The latter has a diaphragm-sealed hole in the cover. The steel melts the diaphragm and enters the casting chamber dissolving into a beam of small droplets offering a very large surface area to the degassing action of the rarefied atmosphere in the chamber. The operational pressure is in the mm. Hg range. By way of example the composition of the gas removed from a chromium molybdenum steel cast into an ingot of 25 tons by the Bochumer Verein process is given as 40% carbon monoxide, 4% carbon dioxide, 35% nitrogen and 23% hydrogen. The effective removal of the latter is the most spectacular feature of the process. Up to 50% of the original hydrogen content has been removed and residual hydrogen contents of less than 2 c.c. per 100 g. have been obtained. In addition other constituents are removed as is shown in the following table. The author proceeds with a discussion of the performance of vacuum cast steel during subsequent fabrication and concludes with brief remarks on the advantages offered by vacuum annealing.

FeO	*MnO*	*CaO*	SiO_2	*Zn*	*Cu*	*Sn*	*Pb*
18%	47%	0.5%	1%	2%	2%	0.2%	1%

Article by L. Colombier *Doc. Metall.* Oct. Nov. Dec. 1956 257-264

Zone Melting

53/I

United States. The term 'zone melting' covers a family of methods for controlling the distribution of soluble impurities in crystalline materials. The principle of the method is that a short molten zone traverses a relatively long solid charge causing re-distribution of solutes in the charge. It was first used to produce germanium of a high degree of purity for transistors but can be used with any crystalline substance that can safely be melted and which exhibits a difference in impurity concentration between liquid and solid state. To metallurgists it is of prime importance as a method of purifying metals. The underlying principle of zone melting is the same as that used in the well-known technique of fractional crystallisation; the considerable increase in efficiency is achieved by melting only part of the charge at one time. 'Zone refining' is the zone melting operation having the object of removal of solutes, *i.e.* purification or separation. As the series of molten zones passes through the charge in one direction the impurities become concentrated at one end of the charge and the remainder is purified, the degree of separation increasing with the number of single zones that pass through the charge. 'Temperature-gradient zone melting' uses extremely small zones which are caused to move by a stationary temperature gradient impressed across the charge. This method can be applied to a joining technique which eliminates the mechanical, thermal or chemical weaknesses of soldered joints; for growing single crystals without a container; measuring and studying diffusion processes in liquids; and to a unique method of making p-n and n-p-n junctions in semi-conductors, that permits conductivity regions of minute size or complex shape to be made inside a semi-conducting crystal. Some of the zone refining methods, combined with vacuum operation, have resulted in the production of some of the purest materials ever obtained, e.g. an impurity of less than 1 in 1^{10}. Perhaps one of the more important applications of zone melting is its contribution to the research on the effects of small concentrations of certain impurities on the behaviour of the more reactive, high-melting metals such as iron, zirconium and titanium. Such studies include mode of allotropic transformation, mechanism of deformation, and the various strength properties.

Article by W. G. Pfann *Metall. Rev.* **2**, 1957 29-76

Vacuum-Melted Alloys Grow

54/I

United States. An outline is given of the type and quantity of products supplied commercially by the chief U.S. producers of vacuum-melted metals. Alloys available include nickel-base and iron-base materials, bearing steels, electronic steels and low-alloy steels. The vacuum-melted metal is characterised by freedom from impurities, improved tensile and impact strength, increased stress rupture strength at high temperatures and special electrical and magnetic properties. Most of the products are used in the aircraft industry for parts such as turbine wheels, shafts and blades, bearings and valves. They are also used in steam turbine parts, atomic energy and nuclear propulsion applications and electronic equipment. Potential future uses are in gas and steam turbines for power plants and in automobiles. Ingots as large as 26 inch. dia. and weighing up to 12,000 lb. can now be produced by the Allegheny Ludlum Steel Corp. Vacuum-melted alloys are priced in the same manner as the common forms of steel. Typical prices quoted are $2.09 per lb. for bars of the iron-base A-286 alloy, $7.775 per lb. for bars of nickel-base M-252, and about $1.30 to $1.45 per lb. for some low alloy and bearing steels. Consumable electrode remelting is expected to prove the most popular form of vacuum-melting. Like induction melting, this method prevents contamination by air or slag. It also gives freedom from contamination by refractories and quick chilling and solidification which are thought to improve the metal structure. Vacuum degassing of ingots is less widely practised. Experiments on the vacuum casting of large forging ingots are being carried out by the U.S. Steel Corp.

Article by Anon. *Steel* 22.7.57 137, 139, 140, 142

Vacuum Treatment of Molten Metal

55/I

Russia. Russian work on the vacuum treatment of liquid steel contained in ladles within large vacuum furnaces has led to the application of the method, on an industrial scale, for transformer and constructional steel. Preliminary trials were carried out at the Enakiev Works where ladle capacities of 16 tons were available with ingot weights of 4 tons. Adequate room must be left in the ladle to allow for the surface agitation of the metal when it boils at low pressure and an initial depth of 200–250 mm. below the top of the ladle was found

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adequate for boiling pressures of 75–140 mm. Hg. The steel was maintained under a vacuum in the ladle for 12–14 mins. and in the mould for 25–30 mins. Samples taken from the resulting ingot showed that in general the steel had a uniform composition and small segregation. The oxygen content was 2.5 to 5 times smaller than that of corresponding non-vacuum treated steel but nitrogen was only reduced by 30–50 per cent. Nitrogen concentration could be further reduced by allowing more intense boiling but it was then necessary to keep the level of the metal 500–700 mm. below the top of the ladle and to lower the pressure to 10–30 mm. Hg. Tests on the low-temperature properties of six specimens vacuum-treated in the ladle showed that the threshold of cold brittleness was −30°C in four specimens and −50°C in the other two. Out of four specimens vacuum-treated in the mould one had a limit of cold brittleness at 0°C, one at −20°C, one under −20°C and one under −30°C. The vacuum process has been applied industrially at the Enakiev Works since 1955, using two furnaces each of 25 tons capacity. Originally the vacuum treatment period was about 10 minutes with a pressure of 60–70 mm. Hg achieved in 5–7 minutes after starting the pumps. Under these conditions transformer and ball-bearing steels are agitated through about 300–400 mm. but the boiling effect is weaker for chrome nickel constructional steel. Supplementary pumping power introduced in December 1955 makes it possible to reduce the pressure to 30–35 mm. Hg. At the Dneprospetstal Works, vacuum-treatment is used for all transformer steel and all chrome nickel constructional steel. The reduction of carbon, sulphur and hydrogen content in transformer steel considerably reduces the watt losses. Trials indicate that the industrial vacuum process reduces the carbon content to not more than 0.02% and the sulphur content to 0.003–0.005%. In the case of chrome nickel steel, vacuum treatment has been found to reduce the number of hair-line cracks in the macrostructure. Work is now being carried out on the vacuum treatment of heat-resistant alloy steels. A foreword to the article says that any desirable effects of vacuum treatment on molten metal can only be preserved by pouring the metal into non-reactive moulds such as dies, ingot moulds or fired-refractory moulds of the investment type.

Article by A. M. Samarin, L. M. Novik, N. I. Gorcharenka, & A. F. Tregubenko *Foundry Tr. J.* 31.10.57 527-529

56/I

Heat-Treating Precision Parts

United States. A method of heat treatment has been developed to be employed for the processing of small precision parts such as screws used in watch making, provided the component is made of hardenable stainless steel. The essential point of the treatment is the proper control of the atmosphere in which the treatment is carried out. Dry hydrogen or dissociated ammonia (both having a dew point less than −60°F) are used for bright hardening and vacuum for high temperature tempering. Annealing is carried out in a dry hydrogen atmosphere and batch assembly under vacuum. All distortion of the component during treatment is eliminated. In fact, if the process is operated under specially selected conditions, the treated parts may be subjected to a controlled growth or shrinkage of their dimensions. The absence of distortion and discoloration makes it possible to finish machining the component prior to treatment and to eliminate all secondary operations such as cleaning and polishing after hardening.

Note by Anon. *Cornell Eng.* November 1956 45

57/I

Glass Resistor for Use in Vacuum Systems

United States. Details are given of a glass resistor, developed by the author for use within a special type of vacuum tube. The physical dimensions of the resistor were not to exceed $\frac{1}{4}$ inch in length and $\frac{1}{8}$ inch in diameter. Glass was chosen as the conducting medium to minimise contamination in the system. A suitable glass-Kovar body developed to support the conducting glass layer is shown in an illustration. The resistance range required of the resistors was 1-10 MΩ. Therefore in view of the design of the resistance body employed the resistivity of the conductive glass had to be 10^6–10^7 ohm. per square. Reference is made to Blodgett's findings that glass with a lead oxide content of 61% would, if reduced in hydrogen, assume resistivity values of about 1,000 megohm. per square. Also the addition of bismuth oxide to the lead oxide reduced the resistivity to a value of 100–1,000 ohm. per square. Accordingly the author chose a glass frit composition containing 61% lead oxide and approximately 25% bismuth trioxide. The preparation of the mixture is described in detail. Similarly details are given of the preparation of the resistor body to receive the glass frit layer. The glass frit was applied either by brushing or spraying. Acetone was used as a vehicle. In the brushing operation the body was rotated in a horizontal position at 75 r.p.m. On completion of the operation the sealing of the frit glass to the body was accomplished by rotating the body in a vertical position and heating it with a low pressure flame of propane-oxygen mixture until red hot. After cooling, the assembly was placed in a small nickel or ceramic boat and passed into a muffle furnace for hydrogen treatment. After heating to 300°C and purging with nitrogen, the hydrogen was admitted to the furnace after circulating through a Deoxo 5–50 unit, flow gauge, molecular sieve dryer and a liquid nitrogen trap. The rate of flow was 1 l./min. and the temperature was varied during the treatment as follows: 300°C for 15 min.; 350°C for 15 min.; 400°C for 15 min. and 450°C for 5 min. There followed cooling down to 100°C in hydrogen and purging with nitrogen during the remaining period of cooling down to room temperature. The completed resistors were tested at 250V d.c., this voltage being used because of the negative temperature coefficient of the resistors and their current dependency on power dissipation. Adjustment to the desired resistance values was obtained by controlled re-oxidation in the form of boiling the resistors in distilled water for a period depending on initial conductivity and layer thickness. The resistors had a black appearance and were unaffected by common solvents. By storing them in the presence of a desiccant all ageing effects were avoided. Outgassing tests, conducted after baking at 300°C, showed no appreciable increase of pressure in the container even at voltages as high as 407V d.c. This involved a power dissipation of 68 mW equivalent to an operating temperature of 152°C in vacuum, although intended only for a power dissipation of 10 mW equivalent to 55°C in vacuum.

Article by L. G. Sloan *Rev. Sci. Instrum.* 27, Dec. 1956 1019-1021

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New Developments in Laboratory Technique at the Leipzig Spring Fair 1957

58/I

Germany. The report includes the description of a short-path distillation apparatus, a vacuum sublimator and a vacuum ballast pump.

Report by E. Krell *Chem. Ing. Tech.* **29**, 1957 661-664

Selected Chapters on Vacuum Technique

59/I

Germany. This collection of short treatises relates to recent work carried out at Messrs. E. Leybold's Nachf. and consists of the following papers: *Further Developments in Molecular Distillation* (E. Keunecke). The deodorising of vegetable oils and the distillation of esters at 10^{-2}–10^{-3} mm. Hg are described and it is shown that colourless products can thus be produced. *Diffusion Measurement of Gases at Low Pressures* (Gunther). The flow of gases at low pressures (10^{-2}–10 mm. Hg) and high speeds is treated theoretically by approximate integration of the Navier-Stokes differential equation. The theoretical values obtained are in good agreement with the experimental results. *Water Adsorption of Condenser Papers as a Function of Temperature and Water Vapour Pressure in the Ambient Space* (H. Schultes). A high-vacuum balance with optical indication permits measurements of moisture contents down to 10^{-2} mm. Hg. The weighing is carried out by regulating the electric current supply to 3 coils, connected in series in a Rayleigh balance. *Development of a Vacuum Gauge for the Range* 10^{-1}–10 *mm. Hg* (K. Ziock). Burning out of the thermo-element at high pressures is prevented in this new type of thermal conductivity vacuum gauge by heating the element indirectly through the ambient air. The calibration curve of the new instrument shows a linear relationship between pressures and deflection of the pointer in the range 0.5–10 mm. Hg. *The Influence of Foreign Deposits on the Structure and Adhesion of Evaporated Films* (E. Giegerl). It is shown that grease, adsorbed gases, esters (*e.g.* in PVC), etc. impair the adhesion and may completely destroy the metallic film. Evaporated intermediate layers may improve the adhesion and homogeneity of the deposit, *e.g.* interlayers of Cu for precious metal deposits or of Cr for optical mirrors or of Ag for Zn layers on condenser papers.

Forschungsber, Wirtschafts & Verkehrsmin. Nordrhein-Westf. **189**, 1955 40 pp.

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Advances in Methods and Applications of Freeze Drying

60/I

Germany. A symposium on problems of freeze drying took place on 8–9th May, 1956 in Cologne at the invitation of Leybold Hochvakuum-Anlagen G.m.b.H. The symposium was attended by 140 representatives from home and abroad. The papers presented and the following discussions gave a good insight into the present state of development and application of freeze drying. This report is a review of the main problems discussed.

Report by K. H. Neumann *Chem. Ing. Tech.* **29**, 1957 267-275

Freezing and Drying Biological Materials

61/I

United Kingdom. Advances in the preservation of biological products are reviewed, covering the past ten years. Particular emphasis is given to the two Symposia on Freezing and Drying, organised by the Institute of Biology in 1951 and in April 1958. Some of the papers read at these two meetings are discussed.

Article by A. V. Smith *Nature* **181**, 21.6.1958 1694-1696

About Freeze Drying of Fruit Juices

62/I

France. Tests have been made in a number of countries to manufacture fruit juice powders by desiccation in a vacuum at low temperatures. The results are good. The powders, when rehydrated, produce fruit juices which are often indistinguishable from fresh juices. The only obstacle to commercial exploitation is the cost of manufacture as the equipment required is expensive. However, the fact that the transport and selling problems of such powders would be much simpler than those of concentrated frozen juices, as used in America, might make the necessary installation worthwhile. To bring the price down to an economic level of, say, 1s. per glass at sales point, very large installations treating tons of fruit a year would be required and must be situated near a source of fruit with an all-year round supply. This seems possible for citrus fruit and perhaps grapes and pineapples.

Article by J. Conan *Fruits d'Outre Mer* **12**, April-May 1957 227-229

13 — ELECTRICAL SCIENCES — 13

Electricity from the Atom

63/I

United Kingdom. Points out that the direct conversion of atomic energy into electricity, without the intermediary of the boiler and steam turbine, is theoretically possible, and describes various experiments which have been carried out to attempt this. In 1913, Moseley collected the beta particles from a well insulated radon source on the plate of a simple form of attracted-disc electrometer, and obtained a p.d. of 150kV. Similar experiments have been made in recent years, using isotope sources. Another recent development of the same idea, is to make a self-charging capacitor with polystyrene (*e.g.*) as dielectric; this allows the beta-particles to pass in one direction, whilst not allowing the bound electrons to leak back. A type occupying 1 cubic inch uses strontium-yttrium-90 and gives 7kV with a short-circuit current of 4×10^{-11} A, an efficiency of 33%. It is estimated that such a cell containing a 10 millicurie source should last 25 years. A different attack on the problem is to bombard a p-n junction in a semiconductor, so that hole-electron pairs are generated in excess

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of those due to the temperature of the junction. This action is like that of the 'Solar Battery', where the pairs are generated by bombardment with photons. Practical cells using Ge or Si have so far produced about one microwatt at an efficiency of nearly 2%. Another type of cell absorbs alpha-particles, producing heat, which then activates a series of thermocouples. In a brief discussion, it is suggested that the semiconductor battery is the most promising, but that the cost of isotopes would have to be much lower before it could be competitive.

Article by J. J. Polling *Discovery* 17, March 1956 111-114

64/I

Maser Oscillators

United States. The maser is a new type of microwave oscillator in which a molecular beam of NH_3 is made to induce oscillation in a resonant cavity. If the cavity is correctly tuned, a stable oscillation is obtained at a frequency determined by a transition between two energy states of the molecule, so that there is a high degree of independence of temperature and pressure. In the experiments described, the beam is directed by focusing electrodes (cooled with liquid N_2), into a flexible, open-ended cavity, $5\frac{1}{4}$ inch. long, tuned by compression. The cavity operated in the TM_{011} mode and had a Q of about 9,000. The whole apparatus was evacuated by a diffusion pump, and a Pirani gauge was used to measure the molecular flux in the beam as it passed out of the cavity. The figure quoted for this flux is 5×10^{14} mole/sec./cm^2. The frequency of oscillation can be made to differ slightly from the molecular frequency, and this was studied by comparing the output of one maser with that of another, using a superheterodyne receiver tuned to 23,870 Mc/s. Graphs present the results of calculations in which it is assumed, alternatively, that all molecules in the beam have the same velocity, or that there is a Maxwellian distribution. For the latter case, the theory predicts a range of values of beam flux over which small changes in flux would not pull the frequency of oscillation. Some data are presented to show variations in frequency and amplitude for beam pressures in the range 0-5 mm. Hg. It is suggested that if the frequency of oscillation is exactly equal to the natural molecular frequency, the oscillator will be stable enough to be used as a frequency standard, and attempts were made to locate this condition by studying the effects on frequency of changes in pressure and in focusing voltage, and of a small magnetic field. The Q of the molecular resonance is about 7×10^6, and it is believed it may be possible to obtain a stability considerably higher than this.

Article by J. C. Helmer *J. Appl. Phys.* 28, Feb. 1957 212-215

65/I

Production of High Temperatures and Nuclear Reactions in a Gas Discharge

See Abstract No.: 34/IV

66/I

On the Initiation of Electrical Breakdown of a High Vacuum Gap

See Abstract No.: 37/IV

67/I

Burning Voltage in Arcs in High Vacuum

United Kingdom. An empirical relationship between arcing voltage and physical properties of the cathode material has been found for high-current arcs between solid electrodes in vacuum. Certain exceptions are significant because, apart from carbon, they are all ferromagnetic metals or alloys. The burning voltage of an arc between two electrodes in vacuum is almost constant, increasing only slightly with increase of current, and depending in the main on the nature of the cathode material. A curve shows arc voltage plotted against the product of boiling point and thermal conductivity for a number of metals and alloys.

Letter by M. P. Reece *Nature* 181, 15.2.1958 475-476

68/I

Criterion for Vacuum Sparking Designed to Include both r.f. and d.c.

United States. An empirical relation is presented that describes a boundary, between no vacuum sparking and possible vacuum sparking. Metal electrodes and r.f. or d.c. voltages are used. The criterion applies to a range of surface gradient, voltage, gap, and frequency that extends over several orders of magnitude. Current due to field emission is considered necessary for sparking, but—in addition—energetic ions are required to initiate a cascade process that increases the emitted currents to the point of sparking.

Article by W. D. Kilpatrick *Rev. Sci. Instrum.* 28, October 1957 824-826

69/I

Electrodeless Discharge Tubes Containing Rare Earth and Heavy Element Halides

See Abstract No.: 119/I

70/I

On the Decomposition of Liquid Hydrocarbons by Small Electric Arcs

See Abstract No.: 177/I

71/I

A New Thermal Image-Converter

United Kingdom. A simple thermal image-converter can be made using the dependence upon temperature of the absorption threshold in a semiconductor. Such an image-converter has been made by using a self-supporting film of amorphous selenium metallised on one side mounted in vacuum at the focus of a parabolic mirror.

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Variations in temperature appear as differences in transmitted intensity, if the film is viewed in monochromatic light at a wavelength near the threshold. If a scene in which there are objects of various temperatures and emissivities is focused on the semiconductor, a visible picture is produced.

Letter by W. R. Harding C. Hilsum & D. C. Northrop *Nature* **181**, 8.3.1958 691-692

Tomorrow's Electronic Light

See Abstract No.: 116/I

72/I

Electroluminescence

(Reference only)

73/I

Article by H. F. Ivey *Westinghouse Engineer* May 1957 93-96

Mercury Atomic Beam Lamp

74/I

United States. The construction of an all-glass, atomic beam emission source using ordinary mercury is described. The unit is a vertical 6 cm. diameter Pyrex tube approximately 5 cm. long, provided with a Dewar flask above its upper end. The lower end is hemispherical and two pairs of short side arms with ground glass flanges carry fused silica windows at the back and front and electron guns at the sides. The windows are sealed with Apiezon wax and the gun assemblies with O-ring seals. The system is evacuated *via* a wide tube at window level. Pumping is carried out by a two-stage mercury diffusion pump using a liquid nitrogen trap. The pump speed is 20 l./s. at 10^{-4} mm. Hg. The lamp mercury supply is contained in a 'crucible' placed 3-4 inch. below the centre of the hemispherical base of the lamp and is connected to it by a collimator tube. This tube is surrounded by liquid nitrogen carried in a Styrofoam container sealed to the crucible with Zeluphen Kitt K, which does not crack at liquid-air temperatures. The base of the Dewar flask above the beam chamber serves as a condenser for mercury vapour. The crucible is heated by an oil bath so as to provide the required mercury vapour pressure. A collimated stream of mercury molecules thus passes vertically upwards along the axis of the beam chamber. The electron guns are designed to produce as little heat as possible without sacrifice of efficiency. Commercial valve cathodes (Silvania 6 CB6) are used. The electrodes are mounted on a brass plate provided with three Kovar seals and cooling water circulates through a 1/16 inch. o.d. nickel tube which forms the grid of the electron gun. Each gun axis is normal to the mercury vapour stream and the stream is thus subjected to intense electron bombardment. The maximum gun emission currents are 400 mA. The optical emission from the bombarded beam of mercury atoms is viewed in a direction normal to their movement. The 5,460 Å and 2,537 Å lines are found to be strongest and high resolution measurements have been made using a spectograph embodying quartz-fluorite optics and a fluorite prism. Fabry-Perot etalons in a double-walled airtight chamber temperature controlled to within 0.02°C, were mounted between prism and collimator. The spectral lines were sufficiently sharp to enable the isolation of the Hg ^{198} component of the 2,537 Å line to be accomplished by the careful selection of interferometer spacers. Thus this line can be studied without using separated isotopes in the source and can be compared with the same line of Hg^{198} produced by a Meggers lamp. It is then found that the present fringes are sharper than those produced by the Meggers lamp but the strong background patterns do not allow quantitative assessment of the spectograms.

Article by R. L. Barger & K. W. Meissner *J. Opt. Soc. Amer.* **48**, January 1958 22-27

Solid-State Light Amplifiers

75/I

United States. A description is given of the different types of solid-state image intensifiers together with a comparison of their methods of operation. The characteristics of photoconductive and electroluminescent materials are summarised with particular emphasis on the problems of their optimum combination in the double-layer type of intensifier with and without optical feedback. The optical and electrical characteristics of the overall devices are compared, and some of the possible applications, such as their use for radar-display and x-ray intensification, are discussed.

(*Authors*)

Article by B. Kazan & F. H. Nicoll *J. Opt. Soc. Amer.* 47, October 1957 887-894

Research at Westinghouse

76/I

United States. A new type of television camera tube, called the "Ebicon" is described briefly. It consists of a layer of selenium which when properly prepared is a satisfactory insulator. On scanning by an electron beam, this layer surface becomes uniformly charged to a potential at which no further electrons from the beam can land on this layer of selenium. Then no signal is received on the lead connected to the opposite side of the selenium layer. If an electron from the photo-surface on which the light image is projected is accelerated and driven into the selenium with high energy, it can produce an avalanche of conducting electrons which flow momentarily and discharge the selenium back surface. When the scanning beam returns to this spot, it recharges the selenium and a current flows in the signal lead. For each high energy electron entering the selenium layer, a hundred or more conduction electrons will flow. It is expected that eventually a camera tube may be developed capable of recording as a distinct flash every photo-electron leaving the surface.

A Note by Anon. *Discovery* **18**, October 1957 413-414

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77/I

Production of Television Tubes

Germany. A report is given of the annual meeting of the Institute of Metals which was held jointly with that of the Deutsche Gesellschaft Für Metallkunde in Western Germany in 1956. Details of the lecture programme are given. In addition the members of the conference visited a number of German factories including the Ulm works for the production of television cathode-ray tubes up to a size of 53 cm. diam. recently opened by the Telefunken Gesellschaft Für Drahtlose Telegraphie m.b.H. When completed the factory will produce 50,000 t.v. tubes a month in three parallel production lines. The total production area is 3,200 sq. m. and the roof is 7½ m. high to keep the working area cool. The transport of the tubes and their treatment is effected by overhead conveyors which descend to working level only for loading and unloading. On arrival the tubes are washed with a 10% hydrofluoric acid solution and subsequently rinsed in water from which all salts have been removed by a special purification plant of 15 c.m. per hour total capacity. The fluorescent screen is deposited by inoculating a suspension of phosphors in waterglass, poured into the tube, which causes a mixture of silicic acid and phosphors to separate out. After drying, part of the internal surface is painted with graphite emulsion in order to eliminate the space charge effects and internal light reflections. At the same time the graphite coating serves as a connection between anode and anode contact. The screen is then aluminised. Prior to the vacuum deposition process a coat of a few micron thickness of lacquer is deposited on the fluorescent layer by means described in detail. Subsequently the tubes pass through a tunnel of 30 m. length where they are heat-treated at 380°C to remove impurities and improve layer adhesion and burn the collodion film under the aluminium. There follows inspection by ultra-violet rays. The tested tubes are fitted with the electrode assembly mounted on a pumping trolley and conveyed through a 20 m. long tunnel where the glass is heated to 400°C and the metal parts degassed by h.f. induction heating while being pumped. Finally the tubes are sealed and the getter is evaporated. On completion of the production the tubes are stored for several weeks, tested again and are only then ready for despatch.

Note by Anon. *Engineer* **202**, 12.10.1956 523-525

78/I

Improved Apparatus for Automatically Vacuum Coating the Interior of Glass Tubes with Metal

United States. An improved form of apparatus for automatically processing the aluminising of cathode-ray tubes is described. It consists of a number of detachable carts which are mounted on an endless movable conveyor which carries them around a stationary frame. Each cart carries a cathode-ray tube which is supported vertically with its neck vacuum-sealed to the pumping-system which is also mounted on the cart and consists of a backing pump and a diffusion pump. Support wires passing through the vacuum-seal carry a heating filament on which the metal to be vaporised is mounted. The various vacuum valves and the filament supply are operated by trip switches which are actuated sequentially by contacts on the stationary frame as the carts are moving round.

A. W. Nelson & J. L. Entwistle Co. *Brit. Pat.* 780,687

79/I

Method and Apparatus for Precipitating Metal from the Vaporous State onto Plates. Particularly for the Production of Selenium Coated Rectifier Plates

See Abstract No.: 73/III

80/I

Rectifying Effects of Sodium Chloride Crystals

Hungary. Gold electrodes were deposited by evaporation *in vacuo*, on to crystals of sodium chloride grown from the melt. Measurements of rectifying properties were made at 250°C, and showed forward to reverse resistance ratios of between 15 : 1 and 30 : 1 for potentials of 100 V. The ratio decreases with increasing potential. A prolonged current in the reverse direction increases the reverse resistance, but a strong forward current decreases the rectification ratio. Differences between electrolytically coloured and uncoloured crystals were observed.

Letter by S. Csaszar *Nature* **181**, 19.4.1958 1158

81/I

Evaporation of Impurities from Semiconductors

United States. Equations are derived for the impurity distribution which arises by evaporation of impurities from the surface of a homogeneously doped semiconductor. The rate of evaporation is assumed to be proportional to the surface concentration. When a rectifying metal contact is made to a semiconductor with such an impurity distribution, the dependence of capacitance on the applied voltage can be used to determine the proportionality constant between rate of evaporation and surface concentration.

(*Authors*)

Article by K. Lehoree, K. Schoeni & R. Zuleeg *J. Appl. Phys.* **28**, April 1957 420-443

82/I

The Metallised Paper Capacitor

Switzerland. The general aim in the design of technical apparatus is to produce components as light as possible and of dimensions as small as is feasible. This applies also to capacitor design where the introduction of new dielectrics such as Teflon and Mylar have helped to reduce dimensions. The latest step in this direction is the introduction of metallised paper for capacitors instead of employing a separate metallic foil. The minimum thickness of a separate foil is 6μ. First to suggest direct metallisation of the dielectric was Mansbridge. His was a chemical method and the quality of the coating was appreciably improved when the metallisation was effected by vacuum evaporation techniques. The thickness of the deposit obtained by this method is of the order of 10^{-5}cm. Zinc, aluminium or, in some cases, nickel is employed for the metallisation of capacitor paper.

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Zinc is the most useful from the angle of the self-healing effect whereas aluminium and nickel show less tendency towards corrosion. The deposition procedure is discussed in great detail and special attention is drawn to the importance of drying the capacitor paper immediately prior to deposition of the metal. Capacitor paper of the type normally employed contains 8% by weight of water. Assuming that the deposition is carried out in a chamber of 100 l. volume at a pressure of 1 μ Hg, 1 g. of water introduced into the chamber would form water vapour taking up a volume of 1,300 c.m. causing the pressure in the chamber to rise from 1 μ to 59 mm. Hg. The size of the vacuum pumps required to remove water vapour at a rate of 1,300 c.m. per sec. for instance would render the process impracticable. The author proceeds to discuss the self-healing effect of vacuum-metallised capacitor paper. The area of the dielectric, exposed by the evaporation of the deposit as a result of a breakdown (short circuit), is of the order of a few sq. mm. An area of 2,800 sq. cm. is required to obtain a capacity of 1 μF. Thus even after about 2,000–3,000 short circuits in vacuum-metallised capacitor paper the capacitance of 1 μF would only be reduced by 1%. It is the self-healing effect which facilitates the reduction of the capacitor dimensions. A short circuit in an aluminium foil capacitor is permanent as the paper normally employed as dielectric always contains small pores promoting short circuits. Thus more than one layer of dielectric must be used whereas the metallised paper condenser requires only one layer for the reasons stated. The procedures employed to facilitate joining the connections to the capacitor are described. The variations of capacitance and loss factor plotted against temperature and frequency are given in graphs. These characteristics are not very different from foil capacitors. Between -20°C and $+70$°C the capacitance varies from -3% to $+2.5$% of its value at 20°C. The temperature coefficient is virtually constant at a value of $5–6 \times 10^{-4}$/°C. The author investigates the Joule losses occurring in the metallised paper capacitor for the case of a.c. operation, and develops an equation for their determination.

Supply Voltage	Specific Volume c.c./μF		$\frac{V_{pm}}{V_f}$ 100%
	Metallised Paper V_{pm}	Foil V_f	
100 V d.c.	1.44	8.0	18
220 V a.c.	5.75	8.0	72
380 V a.c.	15.4	18.7	82.5
500 V. a.c.	23	27.5	83.5

In conclusion, the author compares the volume of metallised paper capacitors with that of foil capacitors. Breakdown resistance depends on the thickness of the dielectric and is calculated on the basis of 30 V per μ thickness, *i.e.* at 8 μ thickness the capacitor can be operated at 240 V. The specific volumes of metallised paper capacitors and foil capacitors are given in a table reproduced below. From this it can be seen that at 100 V the specific volume of the metallised paper capacitor is 1.44 c.c./μF, the condenser being of the single-layer type whereas the corresponding foil capacitor is of the double-layer type containing a foil of 8 μ thickness and a specific volume of 8.0 c.c./μF. The table also shows that there is a smaller difference between the two specific volumes at higher voltages.

Article by A. Kohler
L'Onde Electrique
36, Oct. 1956
842-851

Industrial Power Capacitors

83/I

United Kingdom. The influence of the impregnant on the electrical properties of paper capacitors is discussed. A good vacuum impregnation process is essential to remove included air from the paper and to facilitate flow of the impregnant into the interstices of the material. The size of a capacitor, for a given electrical performance, may be reduced, within certain limits, by increasing the effective permittivity of the dielectric and/or the average dielectric stress in the dielectric. Pure hydrocarbon liquids have been used successfully for many years to impregnate capacitors for a.c. power circuits. Most of these oils have a permittivity of 2–2.3 and, when well dried and evacuated, a dielectric strength at least 20 times higher than that of air. An advantage of a liquid impregnant is the elimination of the shrinkage voids which may occur in a solid impregnant subjected to temperature changes. A newer type of impregnant is a synthetic chlorinated liquid such as pentachlorodiphenyl, which has a permittivity of 5.2 at room temperature, and gives greater security against breakdown of the liquid films. This security is of particular advantage in h.t. networks which are liable to transient voltages caused by atmospheric discharges as well as by switching surges. Pentachlorodiphenyl impregnants require the use of a paper based upon wood pulp and not on linen. A disadvantage is that they cause a greater variation of capacitance with temperature than do oils, and capacitors impregnated with them show rapid rise of dielectric losses at temperatures below 0°C. The latter is due to the change of state to a solid which occurs near this temperature and is a particular disadvantage in outdoor installations which are not operated continuously in very cold weather. Another type of synthetic impregnant, consisting mainly of trichlorodiphenyl, has been introduced to overcome the low-temperature effect. This has a much lower solidifying point so that capacitors can be switched on and off without damage at temperatures at least down to -30°C. The dielectric constant of this material is about 6 at room temperature. Recent researches have shown that under a high electric stress the molecules of an impregnant can be broken down chemically with the liberation of small bubbles of gas. Minute discharges occur in these gas bubbles and result in local heating and further decomposition. Where a mineral oil is used the discharges liberate hydrogen gas and bring about polymerisation of the oil. In capacitors, impregnated with chloridiphenyl, decomposition produces an acid which attacks the paper chemically. A characteristic of the internal discharges is that once established at a certain inception

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voltage, for example by a transient overvoltage, they may persist down to a much lower extinction voltage which may be lower than the normal operating voltage. The discharge inception voltage of trichlorodiphenyl capacitors is higher at normal temperatures than that of pentachlorodiphenyl capacitors, but it falls more rapidly at higher temperatures. The two main types of capacitor are the large tank in which a considerable number of roll capacitor elements, mounted in racks, are grouped in series: and the small unit construction in which only a small number of elements are packed tightly in a container. In the tank type heat-treansfer occurs mainly *via* the thermal circulation of the liquid and the outer tank is heated chiefly in its upper parts. With the unit type heat-transfer takes place over most of the surface area of the container. Units can be mounted or racked up to make capacitor banks of any size.

Article by P. R. Coursey *Elect. Times* 31.1.57 163-167

84/I

Capacitors Having Electrodes Formed of Metallised Dielectric Strips

United Kingdom. A method of constructing small and electrically strong capacitors is described. In previous methods of making capacitors from rolled-up strips of (vacuum deposited) metallised paper, pores in the strip material allow the evaporated metal to penetrate and tend to cause short-circuits. This can be prevented by including an extra sheet of non-metallised dielectric in two layers in the roll, or by lacquering before metallising. The former method increases the bulk to four thicknesses of dielectric per compound layer, *i.e.* to 28 micron, assuming a minimum strip thickness of 7 micron, and the lacquer method makes vacuum metallising difficult owing to degassing of the lacquer in the vacuum chamber. The use is proposed of three strips of dielectric, two metallised without lacquering and one not metallised (but lacquered if necessary). The metallised strips are placed back-to-back to give the desired 'capacitance' with a double thickness of insulant between them, so that pinholes or pores in one strip will be protected by the other, while the insulating strip proper separates the metallised surfaces. This results in a compound layer of three thicknesses, *i.e.* 21 micron and insulation and capacitance properties equivalent or higher than that of four thicknesses, according to the type of material chosen for the non-metallised strip.

Standard Telephones and Cables Ltd. *Brit. Pat.* 758,784

85/I

Miniature Metallised Lacquer Film Capacitors

See Abstract No.: 39/III

86/I

New Capacitor Factory

United Kingdom. Careful control of manufacturing conditions is an important factor in the production of good power capacitors, in order to obtain a high kVA efficiency at a high stress. Consequently, minor faults, due to moisture or dust for example, must be completely avoided. The new power capacitor factory of Johnson and Philips Limited has been designed with these points in mind. Material is tested on arrival, and carefully processed and stored. For example, impregnating oil is filtered, dehydrated and degassed, and stored under vacuum. Processing and assembly are carried out in air-conditioned temperature-controlled rooms. The air is filtered, refrigerated (which removes some moisture) and then warmed to the correct temperature. The air in the rooms is under slight excess pressure to reduce ingress of dust. The wound elements are dried and impregnated under vacuum 12 inch. Trunking is used to connect the vacuum pumps, and the system is so arranged that in the event of a fault occurring the plant is shut down safely, with the vacuum maintained, thus avoiding loss of partially complete items. A comprehensive and rigorous test schedule is followed.

Article by Anon. *Electrical J.* 26.7.1957 260-261 & *Elect. Rev.* 26.7.1957 139-141 & *Elect. Times* 25.7.1957 124-125

87/I

Chromium Nitride Resistors

United States. Evaporated films of chromium nitride or chromium titanium nitride on ceramic bases have been used by the Battelle Institute to form very high stability resistors of small size and large resistance. The temperature coefficient varies with the conditions under which nitriding is carried out and it is believed that a zero coefficient range of resistors may be possible.

Note by Anon. *Wireless World* **63**, Feb. 1957 96 Original Report in *J. Electrochem. Soc.* **102**, Feb. 1955

88/I

Glass Resistor for Use in Vacuum Systems

See Abstract No.: 57/I

89/I

Developments in the Dielectric Drying of High-Voltage Cables

See Abstract No.: 115/III

90/I

Impulse Voltage Strength of Oil Immersed Insulation

See Abstract No.: 108/III

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Dependence of the Dielectric Breakdown of Transformer Oil on Electrode Conditions

91/I

United Kingdom. It is known that the results of tests of the breakdown strength of insulating liquids are not only determined by the properties of the liquid itself but also by the state of the electrodes employed in the tests. The author investigated the behaviour of transformer oil which was filtered, dried and degassed before being placed in a totally enclosed cell where it was subjected to impulse voltages of up to 500 kV of micro-second duration. Stainless steel and aluminium electrodes of a hemispherical shape 2.5 cm. in diameter were used. The gap was 2.5 mm. The electrodes were polished in a manner described in detail prior to each test and then stored in a desiccator with a drying agent until used. After mounting the electrodes in the test cell the latter was evacuated to 10^{-2}mm. Hg before the oil was admitted whereupon the pressure was increased gradually to atmospheric. Early results with stainless steel electrodes were erratic. Upon investigation it was found that the results depended on the storage period of the electrodes. Electrodes not stored at all indicated a breakdown strength of the oil of 1,100 kV/cm. whereas electrodes stored for more than 5 hr. gave results of over 2,000 kV/cm. Aluminium electrodes employed immediately after preparation (cutting out storage) yielded a figure of 890 kV/cm. and aluminium electrodes stored for 15 min. or longer under the conditions described yielded a value of 1,310 kV/cm. In the case of the stainless steel electrodes it was found that the results obtained were independent of the presence of a drying agent in the desiccator. The observations made in these tests are consistent with effects obtained from the oxidation of stainless steel at room temperature and the characteristics of aluminium exposed to atmosphere for 15 min. or more. The presence and action of a natural oxide layer on the metal surfaces of the electrodes employed is interpreted by the author in terms of Mott's suggestions regarding the electrical properties of chemisorbed oxygen on metal surfaces. It can be expected that an electrical double-layer exists on the cathode surface (which is the only electrode affected), the presence of which will reduce the emission of electrons into the liquid.

Letter by R. Hancox *Nature* **178**, 8.12.1956 1305-1306

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Electrostatic Charge Separation at Metal-Insulator Contacts

92/I

See Abstract No.: 35/IV

Improvements r.t. High-Vacuum Electric Circuit-Breakers

93/I

United Kingdom. Alternating current circuit breakers have to interrupt many thousands of amperes in supply lines operating at several kilovolts. Under these conditions current arcs occur between the electrode surfaces of the breaker. The magnitude of the arcing depends on the phase of the current at the time the break is made, and if this is unfavourable the large currents in the arcs can cause damage to the surfaces of the electrodes. This damage is the result of electrode overheating and consequent melting which takes place because the arcs become rooted to the point where they were initiated instead of moving around on the contact surface. Such rooting is avoided in a new high vacuum circuit breaker capable of handling 8,000 A at 11,000 V. The main electrodes of the breaker consist of a pair of axially aligned copper cylinders, the contacting surfaces being the flat ends which are two inches in diameter. One cylinder is fixed and has a centrally recessed tungsten button electrode. The movable cylinder has a central bore in which a subsidiary tungsten electrode is located which mates up with the button electrode and which can move axially independently of the main electrode. When the breaker is closed both pairs of electrodes are held in contact by springs. On the opening of the breaker the main electrodes, which carry most of the current when the circuit is closed, separate before the subsidiary electrodes and small arcs appear between them. The subsidiary tungsten electrodes then open and in so doing break the bulk of the current, heavy arcs being formed between them. The arcing voltage of tungsten is much higher than that of copper and consequently the heavy current arcs are transferred into multiple arcs between the copper electrodes as the tungsten subsidiary electrode retracts into the main one. The cathode spots of these new arcs move about on the cool contact surfaces of the electrodes and no rooting takes place. The contacts, breaking mechanism and operating rod are sealed into a hard glass envelope which is evacuated to 10^{-5}mm. Hg or lower. Movement is effected by means of a stainless steel bellows and the breaking sequence of the main and subsidiary electrodes is controlled by means of a system of springs of different strengths.

E.R.A. Patents Ltd. *Brit. Pat.* 787,846

The Production and Testing of Potted Circuits

94/I

United Kingdom. Details are given of the practical procedure employed in manufacturing small batches of 100 or less potted circuits as applied to the production of a typical electronic unit, a feedback pair, the circuit of which is given in a diagram. Potting of circuits has the disadvantage of adding substantially to the weight of the components and of being inherently of a 'throw away' nature, but nevertheless it has considerable uses, *e.g.* in the case of electronic equipment to be installed in modern aircraft and called upon to withstand appreciable vibration and acceleration forces as well as a variety of temperature and humidity conditions. The following potting material was employed:

	Parts by Weight
Bakelite Polyester Resin SR 17449	100
Catalyst Q 17447	1.66
Accelerator Q 17448	2.25
Filler (200-mesh mica flour)	25

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The filler was added to prevent cracking of the potted blocks at low temperature. At 17°C ambient temperature the setting time was 5 hr. Circuit components such as valves and transformers which are liable to require early replacement, are costly and represent a significant heat source, are not included in the cast block. The remainder is split into sub-units which in turn are split into potted sub-assemblies. The components are mounted between two synthetic resin-bonded paper boards, each punched with a matrix of holes symmetrical in both dimensions, the spacing between the hole centres being 3/16 inch. Tinned eyelets are inserted in one of the two boards (the terminal board) to receive component wires. In addition there are spacing wires which are soldered in with the assembly in a spacing jig after manual adjustment of the position of the components and spacing wires. After having inter-connected the component wires in places as close to the outer surfaces of the boards as possible the free ends of the leads are cropped in a special guillotine. The assembly is now ready for casting. Injection moulds in polythene have been used for the purpose as this material is incompatible with polyester resins and obviates the use of a mould release agent. In most cases the polythene moulds have a common outer profile and the shell and insert of the moulding tool are made of brass. The insert is engraved with the respective component number. Recesses in the mould serve for the location of the assembly to be cast. Immediately prior to casting the assembly is heat-treated at 60°C for 1 hr. to counteract the moisture taken up by the raw edges of the boards which, it was found, had to remain unsealed not to inhibit the setting of the resin. Final preparation of the mixture includes subjecting the latter for a short while to reduced pressure in order to remove air bubbles. As the pot life of a mix is short, moulds have to be filled rapidly. A special filling device is described. If quick setting is desired, release of the air bubbles of the cast block can be accelerated by placing the block in a vacuum chamber. After polymerisation is completed the block is removed from the mould and subjected to a 1-hr. heat-treatment at 60°C in order to harden it further. The component is now ready for use. Due to the nature of the process the terminals in each sub-assembly are closely adjacent to each other. In order to avoid breakdown of the insulation due to humidity in the air and also to provide means for screening the individual sub-assemblies from each other it is advisable to place the sub-assemblies into a metal container, details of which are given. Interconnection is effected by means of miniature Jones plugs and sockets. Valves, etc., are usually placed as close as convenient to the block considering the heat they will dissipate. The article concludes with a description of the special fittings required for production testing.

Article by
T. C. B. Talbot
Electronic Engng.
28, Dec. 1956
512-516

95/I **An Improved Ionisation Gauge Control Circuit**

See Abstract No.: 50/II

96/I **Feedback-Controlled Heat Conductivity Gauge for Measuring Pressure in Vacuum Systems**

United Kingdom. A feedback circuit using a magnetic amplifier is described which enables the hot wire of a Pirani gauge to be maintained very nearly at constant temperature over the pressure range of the instrument. This is achieved by feeding the off-balance current of the associated Wheatstone bridge circuit to the magnetic amplifier in such a way as to alter the bridge current, which is derived from the output of the magnetic amplifier, in the direction which tends to compensate for the change in temperature of the hot wire. Thus, if the gain of the amplifier is sufficient, the bridge will be very nearly restored to its original balanced condition. An Electromethods type 564 magnetic amplifier was used, this being capable of delivering 55 mA into a 200 Ohm load. Graphs are given of the (bridge input current)^2 *versus* pressure and (bridge output current) *versus* pressure, for various wire temperatures and amplifier gains. These show that the true constant temperature characteristic is approached more closely as the amplifier gain is increased, but the discrepancy between the actual and ideal curves becomes considerable at higher pressures because of saturation effects in the amplifier. It is desirable to supply the amplifier from a constant voltage transformer; a 10% change in mains voltage then causes an apparent change of pressure of only 1 μ at low pressures and 3 μ at 500 μ. It is claimed that this circuit has the advantage over the manually-operated constant temperature bridge that it records continuously and that over a wide pressure range the bridge output current is proportioned to pressure. As compared with the constant voltage bridge it has a much greater pressure range for a given maximum wire temperature and a relay can be operated from the bridge supply; the constant voltage bridge is, however, more sensitive in the pressure range 0–50 μ.

Article by
J. H. Leck
C. S. Martin
Rev. Sci. Instrum.
28, February 1957
119-121

97/I **Rhenium Ion Gauge Filament**

See Abstract No.: 51/II

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98/I **Vapour Pressure of Plastic Materials**

See Abstract No.: 107/II

99/I **Evolution of Gas and Permeability to Gas of Constructional Metals for Vacuum Systems**

See Abstract No.: 46/I

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A Graphical Method for the Calculation of Cooler-Condensers

100/I

United States. The method of Colburn and Hougen for the design of cooler-condensers is simplified by the use of a new graphical procedure for the determination of the liquid-gas interface conditions, by which trial and error computations are entirely eliminated. The design method is based on the application of a diagram of the logarithm of the non-condensing gas partial pressure *versus* the temperature. An example is given to illustrate the design method.

(*Author*)

Article by G. H. P. Bras *Chem. Engng. Sci.* **6**, No. 6, 1957 277-282

Metallurgical Research in Nuclear Power Production

101/I

United Kingdom. Metallurgical problems resulting from the behaviour of the materials used in reactor fuels and their containers are discussed under the headings: irradiation stability, thermal stability, sheathing materials, corrosion, fabrication and alloying, and physical properties. The various types of reactor and the special problems of each are outlined. At present, improving the radiation stability of the fuel is an important factor in reducing the cost of nuclear power. The effects of radiation on the crystal structure of uranium and, in particular, the phenomena of wrinkling and 'growth' have been studied. The results indicate that a strong isotropic alloy of uranium, for example a uranium-12 wt% molybdenum alloy, should show good resistance to irradiation damage. Irradiation swelling seems to be due to the accumulation in bubbles of fission-product inert gases. In theory, removal of the gas by diffusion should reduce the effect, but attempts to facilitate diffusion by using loose-sintered powdered compacts have been unsuccessful. Swelling may be mechanically inhibited by enclosing the fuel in a strong can. It may also be counteracted by using special fuel elements. These include elements in which the fissile particles are distributed throughout a non-fissile matrix, cermet-type elements and elements in which there is a non-uniform distribution of alloying element through the cross-section. Ceramic fuels may behave better under irradiation than metals. Liquid fuels should permit easy removal of fission-product gases so that, with these, swelling would be eliminated and nuclear poisoning reduced. It seems unlikely that a suitable liquid alloy of uranium will be found but it may be possible to develop a useful liquid alloy of plutonium. Most canning materials have coefficients of thermal expansion sufficiently different from that of uranium to give rise to "thermal ratcheting" unless special precautions are taken. One precaution would be to place the uranium loose in a can strong enough to resist collapse, but the thickness of can which would be necessary makes this impracticable. The alternative method is to hold the fuel and can so closely together that they behave as a single unit. This is best achieved by metallurgical bonding but mechanical devices may also be used. Methods of bonding which have been investigated include high-temperature duplex rolling and extrusion, powder metallurgy techniques, and casting the fuel into zirconium cans which act as moulds. The ideal sheathing material from a nuclear point of view is beryllium but its application is hampered by production and fabrication difficulties. Corrosion in nuclear-energy work includes corrosion in aqueous, gaseous, liquid-metal and fused-salt media. The development of boiling reactors requires investigations into corrosion under conditions in which both steam and water are present and the gas/liquid interface moves irregularly. A corrosion resistant alloy of zirconium, Zircaloy, has proved a suitable canning material for use in pressure-water reactors, and aluminium-nickel alloys are now being investigated in order to produce a cheaper material for this purpose. Methods of fabricating fuel elements are dominated by the high reactivity of the metals concerned. Processes of interest include 'integral' canning in which the fuel and sheath are completely bonded together, and powder metallurgy, the scope of which will be widened by the development of hydrostatic pressing at high temperatures. Direct extrusion of powders at high temperatures has given good-quality solid metal, and the method has been extended to form cermets by chemical reaction of the components *in situ*. The possibility of processing irradiated fuels by pyro-metallurgical methods may reduce the importance of irradiation stability and may lead to the acceptance of less precise methods of fabrication. Work on the alloying and physical properties of the refractory metals has included a study of diffusion phenomena for various metallic systems. The diffusion of argon in silver has been determined as an analogy to the diffusion of inert-gas fission products in fissile materials. Work is also being carried out on 'slurry' fuels and blankets. Slurries can be made either by cooling an alloy from the liquid to a temperature between the liquidus and solidus of the system or by mixing insoluble particles in a liquid metal. They are of interest because, at practicable temperatures, higher densities of fissile material can be obtained with liquid-type properties than with true solutions.

Paper by J. G. Ball *J. Inst. Metals* **84**, March 1956 239-250

Fabricating and Testing Tubular Fuel Elements

102/I

United States. Thin-walled tubular fuel elements for use in a heterogeneous-reactor mobile-power package are described. The elements are of stainless steel and may give a possible gain of 50% in heat production over the flat-plate elements now in use in reactors. Each element is 25.5 inch. long and has a core containing 20–30 wt% of fully enriched UO_2. The wall thickness including fuel and inner and outer cladding is 30 mils. Similar elements of aluminium are also being developed. The core is formed by consolidating a mixture of UO_2 particles and stainless steel powder to form a tube. This is done by hot-working since the cold-working methods normally used for fabricating tubing give rise to 'stringering' or non-uniform distribution of UO_2. Uniform distribution of the fuel is necessary to prevent concentration of radiation in certain areas with resulting damage to the tube. The tubular core is clad on both surfaces and on its ends to protect the core from the corrosive effect of the coolant and to protect the coolant from the fission products. Direct bonding, in which the cladding material is bonded directly to the core, is used and is preferably carried out in high vacuum. Methods which may be used for bonding include furnace diffusion, cold-pressure welding and liquid-phase welding and brazing. A bonding method which shows great promise is hot pressure welding and this has been investigated for various systems, including Al–Ni, Al–Cu, and Al–Zr. Bond strength increases with increasing pressure and to a certain

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extent with increasing temperature. At a certain temperature level, however, the formation of brittle intermetallic zones results in a decrease of bond strength. Time has a less critical effect than pressure or temperature but with the formation of brittle intermetallic zones, strength tends to decline with excessive holding times. It is important that the parts to be joined should be thoroughly clean. A limitation on this bonding process has been the inability of bonding dies to withstand high pressures at temperature. With Inconel X temperatures up to 650°C and pressures up to 15 psi can be used. Developments in molybdenum alloys have increased the range of the process and dies of the molybdenum–0.5% titanium alloy have been used successfully at temperatures up to 900°C and pressures of 5 psi. Graphite dies cannot normally take pressures above 2–3 psi and might cause contamination. The completed fuel elements must be tested for conformance to specifications. Non-destructive tests which may be used are based on ultrasonics, eddy currents, magnetism, heat transfer, radiography or corrosion. Destructive tests include conventional tube testing methods, peel tests, thermal shock, mechanical shock or metallography tests.

Article by S. Storchheim *Nucleonics* 15, Jan. 1957 85-91

103/I

Fabricating the HRT Core

(Reference only)

Article by W. R. Gall *Nucleonics* 14, Oct. 1956 32-33

104/I

The Welding of Titanium

See Abstract No.: 234/I

105/I

Hard-to-Braze Alloys Behave in Vacuum Set-Up

United States. Vacuum brazing, heat-treatment and degassing have been carried out on an experimental and short-run production scale in a 1,000-lb. vacuum furnace designed and built by the Vac-Hyd Processing Corp. The furnace has been in use over a period of two years without any serious maintenance problem. One object of its use is to extend the applications of high temperature alloys, and metals such as titanium and Inconel have been brazed successfully in the furnace. An advantage of brazing in vacuum rather than in hydrogen is that the possibility of the hydrogen atmosphere failing to penetrate the inner recesses of parts to be joined is eliminated. The vacuum furnace also minimises the possibility of flux not reaching the desired location or of excessive flux causing burnout or corrosion. Less flux is required and problems of flux removal are reduced. Joining is facilitated by the absence of oxide formation on the metal surface. Silver, copper and nickel alloys can be used as bonding materials and the brazing material flows easily in the furnace where temperatures as high as 2,150°F are used. The method makes it possible to braze intricate honeycomb structures and to join very thin sections to thick sections without excessive warpage. Sandwich structures ranging in overall thickness from 0.035 inch. to 0.5 inch. or more have been produced and vacuum-brazed light thin stainless steel or titanium is considered a possible replacement for aluminium in aircraft surfaces requiring high temperature resistance. A potential application of vacuum brazing is in the fabrication of hollow turbine blades. The furnace has also been used to vacuum-anneal automotive instrument parts and to degas gas turbine blades made of titanium. Under vacuum the hydrogen content of such blades is reduced from 200 ppm to less than 50 ppm. A possible application of the furnace is for the simultaneous vacuum coating and brazing of plain carbon steels with nickel base alloy to provide a strong structure with surfaces which are resistant to heat and corrosion. A future application may be in the treatment of metals, *e.g.* springs, which are embrittled by pick-up of hydrogen or other gases. The vacuum retort is 30 inch. i.d. and has a working height of 40 inch. It can be removed from the furnace and pumped down to 25 μ before insertion. Two retorts are available. The required vacuum is obtained by a mechanical pump and an oil diffusion pump. Degassing may be carried out at pressures up to 25 μ but a maximum of 5 μ is usually held during brazing operations. With a 250 to 350 lb. load the furnace temperature can be raised to 2,150°F in about $1\frac{1}{4}$ hrs using resistance-type electric heating elements.

Article by W. G. Patton *Iron Age* 24.1.57 80-82

106/I

Brazing Beryllium Tubing to High-Temperature Alloy Collars

United States. A vacuum furnace brazing technique has been developed for lap brazing beryllium tubes to stainless steel and other high temperature alloys, such as Inconel X and Hastelloy. A gas-tight joint is obtained which can sustain a compressive stress of 4,000 psi at a service temperature of 750°F with the braze itself under shear or bearing stresses. Some tests were carried out on torch brazing and pre-coating by dipping into a molten braze alloy bath, but most of the work was concentrated on vacuum brazing which was found to be the most satisfactory method, despite the more elaborate equipment required. Advantages of vacuum brazing are that it limits the formation of oxide on the beryllium and permits slow heating to and cooling from the brazing temperature, thus preventing thermal shock. It also avoids the need for a highly purified inert gas. Two different vacuum furnaces were used. One consisted of an induction-heated bell jar 6 inch. i.d. by 15 inch. long. This could be heated quickly and was used for brazing simple lap joints in strip material. The second unit was a resistance-heated sintering furnace for beryllium and incorporated a double-shelled vacuum container with a 6 inch. i.d. heating chamber. This was a slower heating furnace and was used for brazing tube assemblies. The lap joint used for joining the tubes was not of the design used in service but was chosen to allow compression shear testing and permit a mechanical evaluation. Most joints were made with the eutectic 72% Ag–28% Cu brazing alloy, because of its relatively low melting point and sharp freezing range. The short brazing times possible with this alloy limited migration of brazing material and thus contributed to joint

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strength. Clean oxide-free surfaces were found to be essential to the production of satisfactory brazes. All parts were first dry machined and then dipped into a 40% HNO_3–2% HF–58% H_2O solution for two minutes. The Ag-Cu braze alloys were cleaned in dilute HNO_3. Test strips were assembled with the braze alloy pre-placed, and a clamping device was used to prevent movement of the components during brazing. A light pressure of about 1 psi was applied to the joint. In the case of tubes, the braze alloy was again pre-placed in the joint and a tight fit was used. A graphite plug was inserted to maintain lap length, butt spacing and axial alignment, and to stop the molten braze alloy from flowing out of the bottom of the joint. Because of the slightly greater thermal expansion of the beryllium inner tube a pressure was exerted on the braze alloy on heating. Maintaining the braze alloy under pressure, in both strip and tube assemblies, prohibited oxide film formation in the joint area during heating to the brazing temperature. Without this precaution it is thought that the oxide film which forms on all exposed surfaces, even in the vacuum of $<1\mu$ used for brazing, would prevent the braze alloy from wetting the base metals. The mechanical pressure also forced out excess molten braze alloy, and this contributed to a stronger joint. In the small, bell-jar furnace, a heating time of about 1 hr. was used to attain the 1,470°F brazing temperature. Samples were held at this temperature momentarily and were then cooled to room temperature in about 2 hrs. In the larger furnace, a heating period of 2 hrs. and a cooling period of 16 hrs. were necessary. The tubes were held at brazing temperature for as short a time as possible in order to minimise diffusion. The brazed joints were examined visually and tensile-shear tests were carried out on the brazed strips and compression-shear tests on the brazed tube assemblies. Most of these tests were carried out at the required operating temperature of 750°F. Metallographic examinations were made and the tubes were also pressure-tested. The joints were found to have shear strengths of about 7,000 to 8,000 psi at 750°F. The joint strength is limited by the strength of the phase adjacent to the beryllium metal. In order to keep this phase as narrow as possible it is advisable to keep the brazing temperature as low as possible to limit the rate of diffusion of copper and silver into the beryllium. A brazing temperature of 1,480°F appears suitable.

Paper by K. G. Wikle & R. Magalski *U.S. Atomic Energy Comm.* Report No. COO-310 June, 1956

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High Energy Nuclear Physics

107/I

United Kingdom. This is a report on the Proceedings of the Sixth Annual Rochester Conference on High Energy Nuclear Physics. One of the most significant discoveries to emerge was the existence of anti-protons, identified among the particles coming from a target bombarded by the proton beam of the 6 billion Volt Berkeley Bevatron.

Article by J. D. Cockcroft *Nature* **180**, 17.8.1957 303-304

Production of High Temperatures and Nuclear Reactions in a Gas Discharge

108/I

See Abstract No.: 34/IV

Zeta

109/I

United Kingdom. A group of three articles describing the Zero Energy Thermonuclear Assembly at Harwell. Special attention is paid to the electronic monitoring and recording equipment. The principle of operation is briefly described: Deuterium gas in a torus of aluminium, 3 m. in diameter and 1 m. in bore is heated to temperatures approaching 5 million deg. C by pulses of current. The gas is made effectively the secondary of a large pulse transformer, through the primary of which is discharged a 1,600 microfarad bank of capacitors charged to 25kV. The discharge, which may carry up to 200,000A peak, is stabilised by a steady magnetic field parallel to the axis of the torus. To obtain steady conditions, it is necessary to repeat the discharge a number of times, while in use discharges are passed every 12 sec., perhaps for many hours. The method of timing the sequence of operations required for each cycle is illustrated diagrammatically. An 8-channel oscillograph, using 4 double-gun cathode-ray tubes is available for recording the results of experiments. Every time a pulse of current is passed through the discharge tube, the four tubes are photographed, together with the date and the serial number of the discharge. The stability of this equipment is such as to permit operation all day without adjustment of brilliance controls, etc. There is also a monitoring unit, with two cathode-ray tubes of a type which store indefinitely the trace (Memotron). The quantities which can be recorded include the outputs of neutrons, and x-rays, spectrographic line intensities, signals from magnetic and Langmuir probes, etc. The circuits of the oscillographic equipment are described in detail, and there is a brief note on the method of controlling the gas pressure within the torus.

Articles by (1) Anon. (2) E. P. Butt (3) A. E. Cawkell & R. Reeves *Electronic Engng.* **30**, March 1958 (1) 108-110 (2) 110-114 (3) 115-120

Electricity from the Atom

110/I

See Abstract No.: 63/I

Reversal of the Parity Law in Nuclear Physics

111/I

United States. A fundamental concept of nuclear physics that has been universally accepted for the past thirty years is that of parity conservation. Basically, this quantum mechanical law holds that two mirror image systems behave identically except for the mirror image effect. Thus two similar radioactive particles

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spinning in opposite directions about the same axis should emit the same intensity of radiation in any given direction if parity is conserved. The behaviour of many subatomic particles produced in recent years by high-energy accelerators has often been inconsistent and Lee and Yound have concluded that conservation of parity is neither supported nor refuted in 'weak interactions' such as K-mesons decay or emission of a beta-particle. Following their suggestions, experiments at the National Bureau of Standards involving the measurements of directional intensity of beta radiation from oriented Cobalt-60 nuclei, carried out at below 0.01 deg. Kelvin, have demonstrated that parity is not conserved in these interactions. A more fundamental unifying theory of the nature of matter and energy may result. The experiments were carried out on Cobalt-60 placed in a 0.002 inch thick surface layer of a single paramagnetic crystal of cerium magnesium nitrate. The crystal was placed in an evacuated flask immersed in liquid helium contained in a Dewar flask surrounded by liquid nitrogen. A thin anthracene crystal placed in the flask served as a scintillation counter for beta-particles, and two sodium iodide counters were used externally to measure the gamma radiation. The cooling to the low temperature (0.003°K.) required for nuclear alignment was accomplished by adiabatic demagnetisation of the paramagnetic salt. The polarisation of the nuclei could be achieved in either direction by reversal of the magnetic field of a surrounding vertical solenoid. It was found that emission of beta particles was greater in the direction opposite to that of the nuclear spin.

Article by Anon. *Engineer* **204**, 19.7.1957 104-105

112/I

Metallurgical Research in Nuclear Power Production

See Abstract No.: 101/I

113/I

The Future for Nuclear Power

United Kingdom. Possible developments in the design of industrial nuclear power stations as a result of experience at Calder Hall are discussed. It is probable that pressure vessels will be spherical in shape, upwards of 60 inch. in dia. and constructed of 3 inch. plate. Difficulties in pressure-vessel design due to advances in operating temperatures may be overcome by the use of double-skin vessels with intercooling so arranged that the outer shell is not subjected to unreasonable temperature. This development in mechanical design will be accompanied by the use of steels having high creep strength, probably low-alloy steels. Coolant gas will possibly be used at about 10 atmospheres pressure. Attempts to improve the heat capacity of the gas by raising the pressure are restricted by the fact that, for a given shell thickness, increased pressure entails the use of a smaller vessel and hence a smaller core with a resulting decrease in output. The heat transfer characteristics of the plant may be improved by using larger mass flows through the cooling channel. Helium appears an attractive alternative cooling gas to carbon dioxide but is available in bulk only from a single overseas source. Hydrogen would be advantageous if it were possible to overcome the difficulties that it forms a pyrophoric compound with uranium at certain temperatures, and has a serious embrittling effect on steel. Improved magnesium alloys should make it possible to use can temperatures up to about 475°C. Beryllium shows promise as a material for cans operated at temperatures above 500°C but it cannot be used to full advantage with the uranium fuel in metallic form. It will probably be necessary to use the fuel in the form of a ceramic or semi-ceramic oxide of uranium. Zirconium is a possible canning material in water-cooled reactors but is unsuitable for use with carbon dioxide. As higher core temperatures are developed it will be necessary to abandon the cylindrical bar of uranium now used, in favour of a small diameter rod, a flat plate or a tubular element. In order to off-set the unfavourable effects of such cores it will be necessary to use slightly enriched fuels which will only be economic when used with high can temperatures. Using a beryllium container and uranium fuel in ceramic form it is thought that a maximum can temperature of about 600°C and a gas outlet temperature of about 500°C will be achieved by 1968. It is probable that steam pressures upwards of 1,000 psi will be used in association with the gas outlet temperatures of 500°C. In the boilers at Calder Hall gas from the reactor passes through a 17½ ft. dia. shell, giving up its heat to tubes which have their surface extended by means of the Babcock stud tube. This arrangement is not likely to be suitable for the higher temperatures and pressures envisaged for future plants. The importance of nuclear stations to firms engaged in power plant production is emphasised by the prediction that, in Great Britain, the proportion of nuclear power stations in new plant constructed by the Electricity Authorities will be 75% by the late 1960's and nearly 100% by the early 1970's. A prediction of the future cost of nuclear power has been prepared from an analysis of present trends in the atomic field, and is compared with a similar forecast for the cost of power from conventional stations. Electric power from the first industrial nuclear stations which will be completed in 1960/61 is expected to be slightly dearer than that from conventional stations completed at that date. By 1970 nuclear power will be appreciably cheaper and its cost advantage will increase in later years.

Axel Johnson Lecture by C. Hinton *Engineer* **203**, 22.3.57 442-444

114/I

Pilot Plant Generation of Fission Products in Uranium-Bismuth Reactor Fuel

United States. One purpose of the work described was to obtain operating experience with, and to test the components of, a pilot-scale 'loop' which represented the first attempt at handling a U-Bi fuel containing live fission products under conditions approaching those which would exist in a commercial plant. In operation the bismuth, containing 800 p.p.m. natural uranium, flowed through the in-pile section where fission products were generated; thence to a de-gassing vessel to remove volatile fission products; to a contactor where chlorides were extracted with a fused-salt mixture; and back to the in-pile section. Parts in contact with bismuth and fused salt were made from Type 347 stainless steel. The flow rate was 0.25 gals/min. at a temperature of 500°C. The loop gas system consisted of a purified helium supply, a high vacuum system and a low vacuum system. The experimental programme of metal-gas and metal-salt equilibrium studies was started after operating the

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equipment for ten days to permit the build-up of activity in the loop. This work continued for 4,700 hours before one of the electro-magnetic pumps developed a leak but the operation was still continued (with careful checks against spread of alpha contamination) until, after 6,446 hours the sump tank heaters failed and the loop was dumped. Prior to the leakage no difficulty was experienced with the volatile fission products. There was some plugging of the salt lines when using fused chlorides of lithium and potassium but this cleared rapidly when magnesium chloride was added. There was no contamination of the loop by oxygen as shown by the constant concentration of uranium. The enclosure and exhaust system contained the alpha contamination successfully after the leak and no build-up in activity occurred outside the enclosures. The experiment showed that polonium can be controlled when it is present as a dilute bismuth solution without undue hazard. The only modification shown to be necessary was in the design and mounting of the pumps in the loop.

Article by C. J. Raseman H. Susskind C. H. Waide *Chem. Engng. Prog.* **53**, Feb. 1957 86F-92F

Molecular Beams

115/I

United Kingdom. Using molecular beams, individual neutral molecules can be studied free from interaction with their neighbours. They have been used to verify the fundamental postulates of the kinetic theory of gases, to give proof of the wave character of moving particles, to establish the principle of space-quantisation, to determine precisely nuclear magnetic moments, to determine the Lamb shift in the fine structure of atomic hydrogen, and have been applied in the atomic beam clock which is capable of measuring time intervals with an accuracy of 1 in 10^{13}. To produce a molecular beam, the pressure of gas or vapour in a container with a small exit aperture is adjusted so that comparatively few molecules escape into an attached collimating chamber which is evacuated to a pressure sufficiently low for the mean free path of the molecules to be large compared with the distance from the aperture to the walls. Dunoyer made the first molecular beam experiment using sodium in 1911. Stern *et al.* in 1920 first measured accurately the thermal velocity of silver atoms in a beam and the velocity distribution of the molecules in a gas in thermal equilibrium with its surroundings. The usual technique employs a small chamber containing a gas or vapour at a few mm. of mercury pressure and with an exit slit commonly about 1 cm. × 0.02 mm. The conditions necessary to ensure true molecular effusion are discussed, and the angular distribution of the beam is considered in relation to the pioneer work of Knudsen and of Clausing. Brief accounts are given with notes on sensitivity, application, advantages and disadvantages of following types of detectors for molecular beams: Condensation targets (including methods of intensification and the use of beams of radioactive atoms); colour change devices for chemically active beams, bolometers, surface ionisation detectors, Stern-Pirani manometers; electron bombardment detectors; space charge devices; a mass spectrometer with an oscillating electron ion-source and an electron-multiplier positive ion detector capable of measuring the intensities of beams of all kinds of molecules. The various magnetic field arrangements used in the deflection experiments for determining the magnetic properties of free atoms and molecules are discussed. Included are the wedge-shaped pole-piece (Stern and Gerlach), the flat pole arrangement (Rabi) and the two-wire field (Rabi, Kellogg and Zacharias). Classical experiments outlined include the Stern-Gerlach experiment on the discrete orientations of the magnetic moment vector of silver atoms in a magnetic field, the determination by Stern *et al* of the magnetic moment of the proton and the deuteron and those of Stern and Frisch with pure parahydrogen. The fundamental physical significance of these experimental results is considered, leading to the conclusion that the magnetic moments of the proton (+ 2.792743 nuclear magnetons), the neutron (− 1.913139 nuclear magnetons) and the deuteron (+ 0.8574073 nuclear magnetons) are not strictly additive. The molecular beam magnetic resonance method (Rabi) is described. It enables studies to be made of metastable and excited states of molecules and the sign of the nuclear magnetic moment. High precision in measurement is possible as the frequency determination necessary can be very accurate though precision measurement of magnetic field strength is difficult. But the latter problem is simplified, once a particular magnetic moment has been determined absolutely because its resonant frequency can be used to calibrate the field absolutely. Molecular beams are also used in the exploration of oxidation and polymerisation reactions, fluorescence and phosphorence phenomena. Particularly interesting in field of research on hydrocarbons. Recently introduced electric resonance method allows observations of transitions between particular rotational states of polar molecules. Electric dipole moments and moments of inertia of molecules can be determined. Possible future applications are the study of the mechanism of adsorption and of friction.

Article by R. Schnurmann *Research* **10**, 1957 185-197

Tomorrow's Electronic Light

116/I

United States. The electroluminescence of a phosphor as distinct from fluorescence induced by x-ray, cathode ray and ultra-violet light radiation of the phosphor was first discovered by Destriau in 1947. An electroluminescent cell consists of a glass plate to which a transparent conducting coating of suitable material is applied. A phosphor powder-binder mixture is sprayed onto the conducting coating and in turn is coated by an evaporated aluminium film which acts as the second electrical contact. The emission of light depends on the frequency applied. Red phosphor, for instance, gives no light at all at low frequencies. Other phosphors change their colour as a function of the applied frequency, *e.g.* green light at 60 c./sec. and 600 V and blue light at 10 kc./sec. The cells, being completely transparent, can be stacked and by changing the frequency each cell can be excited one after the other by a resonant circuit. Thus at 8kc./sec. blue light can be obtained, and at a slightly higher frequency a green light, and at 18 kc./sec. red light from the assembly. This arrangement could be employed for traffic signals. Transistor oscillator circuits are used as a source of variable frequency power. The degree of brightness obtained varies. One cell at 60 c./sec. and 110 V will give a brightness of $2\frac{1}{2}$ foot lamberts whereas another at 60 c./sec. and 220 V will give 8–10 foot lamberts. The latter figure is about one quarter of the brightness of a normal television screen. These figures have been considerably improved in recent years and a brightness of 2,000 foot lambert has been reported which is higher than the surface brightness of a 40W fluorescent tube.

Note by A. A. McKenzie *Electronics* **29**, Nov. 1956 190, 192

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Electroluminescence

United Kingdom. The type of electroluminescence considered is that produced in zinc or cadmium sulphide or selenides activated with copper. The phosphor is suspended in a suitable dielectric medium such as silicone resin on a supporting sheet of mica and the electric field is applied between conducting surfaces close, and parallel, to the resin sheet. The front conducting surface must be transparent and is usually formed by sputtered cadmium oxide or stannic oxide deposited from the vapour phase. The phosphor layer is backed by a film of aluminium which forms the second conducting surface and also serves as a reflector. Complete design details of the device are given. Alternating voltages up to 1,500 V may be used with these panels but at 230 V 50 c/s the efficiency is similar to that of commercially available panels. The only limit to applied voltage and hence to brightness is the breakdown strength of the dielectric layer. Use of such sources depends on their thinness and on negligible heat dissipation. Several references to the use of such panels are given and briefly discussed.

Article by D. W. G. Ballentyne *Wireless World* **63**, March 1957 128-132

118/I

A Device for Tensile Tests of Irradiated Metals

See Abstract No.: 202/I

119/I

Electrodeless Discharge Tubes Containing Rare Earth and Heavy Element Halides

United States. A method is described for the preparation of electrodeless discharge tubes containing very pure rare earth and heavy element halides to be used as light sources in spectrographic investigations. This type of source is particularly advantageous when the material under investigation is either extremely scarce, radioactive, or both. Tubes have been prepared with as little as 0.1 microgram of sample, and containing as much as 2 curies of radioactivity. The method described is designed to avoid the presence of impurities, whether liberated from the walls or introduced with the sample. It has been found that the most suitable halide is the iodide and this is prepared by reacting aluminium iodide with the sample oxide. The latter is obtained by precipitating either the hydroxide or oxalate from an acid solution; it is then centrifuged, washed, dried and ignited to convert it to oxide. It is immediately placed in a reaction tube and sealed to the vacuum line. Aluminium iodide is prepared by heating aluminium and iodine crystals in a vacuum-sealed quartz tube at 200–250°C. The reagent tube is slowly sealed to the vacuum line. After pumping down, the reagent tube capillary is broken by iron slugs in glass and the tube moved (with the aid of an iron slug and external magnet) so that the tip of the capillary protrudes into the reaction tube. On heating the reagent tube with a furnace, the aluminium iodide is distilled into the reaction tube which is then sealed off. The sample iodide is produced by heating the reaction tube in a furnace at 500°C for eight hours. The sample iodide is then transferred to the discharge tube by a repetition of the slow procedure after thoroughly outgassing the latter at 1,100°C for two hours. The system is then filled with neon, a discharge passed and the neon pumped away; this procedure is repeated until the neon spectrum, as observed in a hand spectroscope, remains pure. The discharge tube is then sealed off and placed in a special furnace in which the temperature can be raised to 750–800°C, at which temperature the vapour pressure of the sample iodide is sufficient for the discharge to be excited by a microwave generator operating at 2,450 Mc/s. Successfully tubes have been prepared using *inter alia* Pm, U and Am. By adjusting the operating temperature it is possible to change the sample vapour pressure over a range sufficient to give an appreciable differentiation between the arc and spark spectrum of these elements. Representative spectra are shown.

Article by F. S. Tomkins M. Fred *J. Opt. Soc. Amer.* **47**, December 1957 1087-1091

120/I

Differential Sputtering of Grain Boundaries in Polycrystalline Tin

See Abstract No.: 94/III

121/I

The Condensation of Iron and Platinum on Surfaces of Single Tungsten Crystals at Elevated Temperatures in The Field Emission Microscope

See Abstract No.: 59/II

122/I

The Effect of Thin Films of Gold on the Mechanical Properties of Cadmium Single Crystals

See Abstract No.: 95/III

123/I

Methods and Results of Modern Single Crystal Growing II. Special Growing Processes

Germany. Details are given of a paper presented to the Meeting of the German Production Engineers' Association at Munich on October 3–5, 1955, dealing with the following methods: growing of crystals from solution, the hydro-thermal method, growing from the melt, crucible-free methods and growing from the gaseous phase. Each method is described in detail and examples are given. Various types of furnace for the growing from the melt are illustrated, such as the Stockbarger, Strong and Kyropoulos constructions. The latter was used at the laboratories of E. Leitz at Wetzlar to produce a KCl crystal 25 × 25 × 25 cm. The methods of crucible-free crystal growing are exemplified by the Verneuil flame-fusion process using a 50 c/s electromagnetic vibrator, and the Pintsch process for the production of monocrystalline fibres and wires of refraction metals such as Mo

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and W; zone-refining methods are also included in this survey, but the description is restricted to the production of very pure Ge. The only references to vacuum methods are the Stockbarger furnace for growing LiF and CaF_2 crystals in argon or nitrogen, and a very brief mention of the Straumanis sublimation process for growing idiomorphous Zn and Cd monocrystals. 112 References.

Article by A. Neuhaus *Chem. Ing. Tech.* **28**, May 1956 350-365

Measuring Flux Absolutely with Indium Foils

See Abstract No.: 90/II

124/I

Determination of the Surface Tensions of Molten Lead, Tin, and Indium by an Improved Capillary Method

125/I

United Kingdom. The method is based on the relation between the surface tension of the metal and the difference in the pressures required to raise the molten metal from a crucible to an equivalent level in two capillaries of different bore. It is suitable for use up to temperatures of about 1,000°C. If the two pressures, in the tube of radii r_1 and r_2 respectively, are indicated by heights h_1 and h_2 cm. on a manometer containing fluid of φ_s gm./cm³ then it is shown that:

$$\gamma \cos\theta = -\frac{g\,\varphi_s\,(h_1-h_2)}{2\quad(1/r_1-1/r_2)}$$

where γ dynes/cm. is the surface tension and θ is the contact angle between the liquid metal and the capillary wall. A subsidiary experiment shows $\theta > 170°$ for lead, tin and indium so that $\cos\theta$ may be taken as -1 with little error. The capillaries are of fused silica and are mounted vertically in a brass block with their lower ends approximately level. A rack and pinion allows for height adjustment. Each capillary contains a molybdenum contact wire the position of which is controlled by a screw thread. The brass block is sealed into a brass cylinder which fits into the top of a 3-inch. dia. silica furnace tube wound with "Brightray C" resistance wire. A silica crucible supported concentrically below the capillaries holds the metal. The contact wires are levelled by first lowering them into contact with the melt. They are then withdrawn, with no relative movement, a distance of 3–4 mm. back into the capillary and the wire in the larger capillary is further withdrawn by the amount necessary to compensate for the meniscus. The capillary and furnace system is pumped by an Edwards 1½-inch oil diffusion pump, backed by a two-stage rotary pump. The overall pumping speed is 6–7 l./sec. and the best vacuum obtainable is about 4×10^{-5} cm. Hg with a leak rate of less than 3×10^{-5} cm./min. Pressure measurements from 76 to 10^{-1} cm. Hg are made by a mercury manometer, from 10^{-1} to 10^{-4} cm. Hg by a McLeod type gauge and below 10^{-4} cm. by an ion gauge. Hydrogen is introduced *via* a purification train. The furnace is first outgassed and is flushed with hydrogen while being brought to the precise temperature required. The furnace pressure is then adjusted to 55–60 cm. Hg, the contact wires are levelled and the capillaries are immersed in the melt to a depth of 1–2 cm. When thermal equilibrium is established, one arm of a silicone manometer is connected to the main gas space in the furnace and the other arm is connected in turn to each capillary. Pressure in the capillary is slowly reduced by means of a needle leak valve until metal enters and contacts the wire. The manometer is then read, after which the metal is blown out of the capillary and hydrogen bubbled through the melt. When sufficient readings have been obtained the capillaries are withdrawn from the melt, the furnace is evacuated and then re-filled with hydrogen while being heated to the next temperature. The surface tensions, γ, obtained for lead, tin, and indium at temperature T°K from the m.p. to a few hundred °C above it are given by:

γPb	=	624—0.24T dynes/cm.	γPb327°C	=	480 dynes/cm.
γSn	=	599—0.065T dynes/cm.	γSn232°C	=	566 dynes/cm.
γIn	=	602—0.10T dynes/cm.	γIr156°C	=	559 dynes/cm.

These values are higher than the most reliable previous values, possibly because of the high-purity metal used. The results lead to molar surface enthalpies of about 9,600 cal. for lead, 8,500 for tin and 8,300 for indium and molar surface entropies of, respectively, about 3.3, 0.4 and 0.8 cal./°K. These values are slightly temperature dependent, leading to positive values of the molar surface heat capacity in each case. They are compared to bulk thermodynamic magnitudes in a discussion of surface bonding and mobility.

Paper by D. A. Melford & T. P. Hoar *J. Inst. Met.* **85**, Jan. 1957 197-205

A Determination of the Surface Tension of Liquid Tungsten by the Drop-Weight Method

126/I

United Kingdom. The production of molten drops by bombarding the lower end of a vertically suspended rod with electrons has enabled the drop-weight method to be applied to the measurement of the surface tension γ of liquid tungsten. The mean value obtained is 2300 dyn cm^{-1} at the melting temperature of 3,380°C in a vacuum of approximately 10^{-4} mb.

(*Author*)

Paper by A. Calverley *Proc. Phys. Soc.* **70B**, 1957 1040-1044

Low Temperature Physics in the U.S.S.R.

(Reference only)

127/I

Article by K. Mendelssohn *Nature* **180**, 7.9.1957 460-462

Physics Below 1°K

United Kingdom. A temperature T can only be called low if kT is small compared with some energy level spacing characteristic of the system. On this criterion 1°K is often not a low temperature, and the region

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below 1°K is one in which many events occur. The adiabatic demagnetisation of paramagnetic bodies is the commonest technique for attaining these low temperatures, though other methods also exist. The main limitation on experiments is the small amount of power which can be absorbed by the working substances. At temperatures down to 3×10^{-2}°K, a number of experiments have been performed, not only on the refrigerants but also on other substances. Results from several branches of physics including solid state and nuclear physics are briefly presented and discussed. Below 3×10^{-2} °K only the refrigerants have been investigated: the lowest temperature yet reached is about 2×10^{-5} °K.

(*Author*)

Article by E. Mendoza *Brit. J. Appl. Phys.* **8**, June 1957 219-228

129/I

The Magnetic Refrigerator for Temperatures below 1°K

United States. The magnetic refrigerator is a device for maintaining isothermally a reservoir and/or experimental apparatus at temperatures below 1°K. Starting from temperatures maintained by a liquid helium bath the device produces cold by the magnetic cooling method, the cooling being performed in a cyclic manner. By cyclic operation temperatures below 1°K, as low as approximately 0.2°K, can be maintained continuously. In this paper some design problems are considered in detail and a comparison is made between the theoretically computed performance and the experimental observations on the first few magnetic refrigerators which have so far been constructed. The 'engine' of the magnetic refrigerator basically requires, besides a paramagnetic working substance for the magnetic cooling process, thermal valves which may allow or block the flow of heat between the working substance, the helium bath and the low temperature reservoir. In the work reported in this paper the material for the working substance has been ferric alum and the thermal valves have been constructed from superconducting lead wires. A discussion is given of the requirements for an ideal working substance and experimental results are reported on some unusual paramagnetic materials which have been investigated for use both as working substances and for reservoir materials. Amongst these ferric acetylacetonate and synthetic ruby have been found to show useful properties. Finally, some of the possible uses of the magnetic refrigerator are considered, as for example in calorimetry below 1°K, for the establishment of an isothermal reservoir for the initiation of single-stage magnetic cooling, for the production of liquid helium baths below 1°K and for the possible subsequent extension of the magnetic refrigerator to a two-stage device for the production of isothermal temperatures well below 0.1°K.

(*Author*)

Paper by J. G. Daunt *Proc. Phys. Soc.* **70B**, 1957 641-652

130/I

Thermal Contact and Insulation below 1°K

United States. The paper reports on a methodic study of single paramagnetic crystals with a view to establishing adequate criteria for the design of experimental sample supports yielding reproducibly small heat leaks. Single crystals were used because, if comparatively free from gross imperfections, they rapidly attain thermal equilibrium. The information gathered from the experiments described was used to construct a support and determine the optimum initial exchange gas conditions and pumping time which would give a minimum heat leak.

Article by J. C. Wheatley, D. F. Griffing & T. L. Estle *Rev. Sci. Instrum.* **27**, Dec. 1956 1070-1077

131/I

Vacuum-Insulated Transfer Tube

See Abstract No.: 188/III

132/I

Properties of Materials at Low Temperatures

United States. This is the last of three articles on low temperature and deals with thermal insulation and the mechanical properties of solids.

Article by R. J. Corruccini *Chem. Engng. Progr.* **53**, Aug. 1957 397-402

133/I

An Improved Inductance Method of Measuring Susceptibilities of Small Paramagnetic Specimens at Low Temperature

United Kingdom. Paramagnetic susceptibilities of rigidly located specimens are usually measured by mutual inductance methods at liquid helium temperatures but at higher temperatures such measurements are subject to large errors. The experiments described avoid many of these limitations by arranging to be able to move the specimen during the course of the experiment. A value for the total susceptibility is first obtained with the specimen removed and a second measurement made after replacing the specimen. The interval between the two readings can be very short thus eliminating several sources of error. The specimen which must not be electrically conducting because of eddy currents, has a maximum diameter of 5 mm. and is enclosed in a Teflon container which is screwed to the end of a long Tufnol rod of 3 mm. in dia. This rod is enclosed in a glass tube and the free end of the rod emerges *via* a Wilson seal. The glass tube is filled with low pressure helium to maintain thermal equilibrium. The specimen position is controlled by raising or lowering the rod by a suitable mechanism. Inductance coils are wound on the outside of the centre glass tube. The secondary coil is wound in two opposing halves to reduce the residual mutual inductance and these halves are matched before use. A Dewar flask surrounds the centre glass tube and contains liquid helium or hydrogen. The specimen temperatures are inferred from vapour pressure measurements. The flask is attached to the main apparatus by a ground glass cone fitting into a copper socket and sealed when cold with glycerol. The entire cryostat is surrounded by a second Dewar vessel containing liquid hydrogen or liquid nitrogen. Two small

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spring clips are attached to the Tufnol rod where it passes below the surface of the coolant and these make good thermal contact with a short length of German silver tube forming part of the centre glass tube. This prevents heat entry along the Tufnol rod. Measurements are made using a Hartshorn Bridge at 175 c/s. The sensitivity and reproducibility of the apparatus are such that valves of susceptibility of 3×10^{-6} c.g.s. units can be made with an accuracy of 1%. Calibration procedure and sources of error are discussed in detail.

Article by F. R. McKim & W. P. Wolf *J. Sci. Instrum.* **34**, February 1957 64-67

Simple Equipment for a Small Low Temperature Laboratory

134/I

United Kingdom. Since the establishment of the Liquid Helium Pool (see E. Mendoza, *Vacuum* 4, Oct. 1954, 476–485) it has become practicable for small laboratories to undertake low temperature work. The authors describe simple, cheap equipment for use in a laboratory not primarily concerned with low temperature phenomena. The apparatus is versatile, enabling a wide variety of experiments to be performed at temperatures between 1.3° and 33°K, and uses liquid helium and liquid oxygen bought from the N.P.L. Provision for recovering the helium gas in a pure state is included. The liquid helium is delivered and stored in a double Dewar vessel, using liquid oxygen between the two shells. Evaporated helium from the storage vessel and the low temperature system is fed to a low pressure gas holder (rubberised fabric cylinders), and later pumped into high pressure cylinders. The cryostat is also a double Dewar vessel using liquid oxygen, the insulating shell of the inner vessel being connected to a rotary pump *via* a tap. This enables the contents of the inner vessel to be pre-cooled by admitting air between the walls of the vessel, which then gives thermal contact with the liquid oxygen. A good vacuum could not be maintained here owing to diffusion of helium through the glass of the vessel, so that connection to a pump is an advantage. The specimen is contained in a copper calorimeter mounted on a stainless steel tube connected to the lid which is sealed with an O–ring. Liquid helium is syphoned into the cryostat *via* a vacuum-jacketed tube. Pressure, built up in the storage vessel by evaporation of liquid helium, is used to operate the transfer. Temperatures below 4.2°K are obtained by lowering the pressure in the cryostat. This is done using a rotary pump, the pressure being reduced to a minimum of 1.5 mm. Hg. Either manual or automatic pressure control can be used. Temperatures above 4.2°K are obtained using carbon desorption. Helium is adsorbed by activated charcoal until saturated. Desorption of the helium gas can then be controlled to balance the temperature rise of the cryostat. Below 4.2°K, temperature is measured by observation of the vapour pressure of helium using a simple mercury manometer. Above 4.2°K, a gas thermometer is used. Two pieces of auxiliary apparatus described are the liquid helium level indicator and the helium gas purity tester. The first uses thermal oscillations set up by the temperature gradient created in a hollow dipstick introduced into the storage vessel. The second measures the vapour pressure of the impurities which are obtained by freezing them out from the helium gas by inserting a sample into the liquid helium.

Article by R. F. Broom & A. C. Rose-Innes *J. Sci. Instrum.* **33**, Nov. 1956 420-424

Storage of Free Radicals at Low Temperature

135/I

See Abstract No.: 181/I

A Preliminary Study of Some Aspects of a Heavy Water Plant

136/I

See Abstract No.: 172/III

The Freezing of Water Droplets

137/I

South Africa. The need for information on the factors governing the freezing of cloud droplets has been stimulated by experiments to control precipitation artificially. In the experiments described droplets were allowed to form on the lower end of a cylindrical metal block inserted into a Perspex holder which was mounted on the stage of a metallurgical microscope. Droplets of the required size were maintained for long periods by covering them with a film of silicone oil and a glass cover slip with a mica spacer, allowing the same droplet to be frozen and melted many times. The metal block was cooled by conduction using liquid oxygen or a dry ice-acetone mixture in a container on top of the block. Cooling and heating were controlled by an electric heater placed between the metal block and the cooling agent. The condensation of moisture on the outside surface of the cover slip was prevented by maintaining an atmosphere of dry oxygen in this region. Six different surfaces were prepared on the highly polished lower end of the block on which the droplets were formed: Collodion, silicone, electrolytically polished stainless steel, nickel, gold, and silver. The last two were obtained by evaporating, *in vacuo*, these metals on to brass blocks. Observations of the freezing of some hundreds of droplets 10 to 20μ in diameter showed that while all the surfaces investigated did possess regions which stimulated freezing, and despite the wide variation in distribution of freezing temperatures, the lowest freezing temperatures were independent of the nature of the surface. Investigation of individual droplets showed that they could be classified into three groups: Those freezing consistently within about 1° of the lowest temperature obtained, those having freezing temperatures spread over several degrees but occasionally reaching the lowest temperature; and those which always froze above the minimum. The majority of droplets on a freshly prepared silicone surface were of the 'lowest freezing' type. A ten-fold increase in the cooling rate reduced the temperature of any given probability of freezing, on lowest freezing droplets, by 0.5°C. Droplets of other types behaved in a more erratic and unpredictable manner which probably accounts for the different value obtained by earlier workers. With a volume increase of the droplet of 50 times, the reduction of super-cooling was observed to be just over 1°, and with a volume increase of three times it was 0.3°. The results of this investigation, taken in conjunction with the results of other workers, seem to indicate that freezing is a statistical process. It is

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concluded that spontaneous freezing temperatures are little influenced by the volume of the droplets, and within the range of sizes of cloud droplets a million-fold increase in volume will raise the temperature for a given probability of freezing by only 3°C. From the conclusion that the results obtained apply to spontaneous freezing of water, the surface free energy at an ice-water interface was calculated. Applying this result to the theory of homogeneous nucleation leads to the conclusion that there is no sudden large increase in nucleation rate in the vicinity of the well-known transition region of water droplets near −40°C which would make this region a barrier to further super-cooling. Lower temperatures could be attained with smaller droplets or more rapid cooling.

Article by A. E. Carte *Proc. Phys. Soc.* **69B**, Oct. 1956 1028-1037

138/I

The Mechanism of Water Vapour Movement in Porous Materials

See Abstract No.: 10/**IV**

139/I

An Experimental Study of the Effect of Speed and Composition of a Steam and Air Mixture and of the Degree of Vacuum on Heat Emission during the Condensation of Water Vapour

Russia. In these experiments a steam and air mixture flowed from top to bottom through a set of cupronickel tubes. The set consisted of 25 tubes, of 16 mm. o.d. and 1,500 mm. active length, arranged in a checkerboard pattern with 10 horizontal rows. Special measurements were made in the middle tubes of the first, third and tenth rows. In each of these the temperature was measured at 9 points by copper-constantan thermocouples. Differential pressure gauges measured the water flow and the rise in temperature was measured by differential thermocouples. In addition, measurements were made of the initial and final temperature of the steam and air mixture, the absolute pressure and the total flow of water, steam and air. As a result of the series of experiments it was concluded that: (1) The effect of the velocity of steam and of the mixture on the coefficient of heat emission was proportional to $(Y_m W_m^2)^{0.16}$ where Y_m = specific gravity of the steam and air mixture (kg./m³) and W_m = velocity of the steam and air mixture in a narrow section of a tube (m./sec.) (2) During motion of the mixture the air with the steam had no undesirable effect on the coefficient of heat emission up to partial pressures of air of 1% (1.5% of the weight of the steam). (3) The effect of the air content in the steam on the coefficient of heat emission for a high vacuum (absolute pressure 30 mm.) is proportional to $e^{-\frac{7PA}{PM}}$ and for a lower vacuum (absolute pressure >70 mm.) it is $\left(\frac{PA}{PM}\right)^{-0.14}$ where PA/PM = air partial pressure in the mixture. (4) The negative effect of the thermal stress on the coefficient of heat emission does not apply for small thermal stress, the coefficient becoming constant at thermal stress <20,000 kcal./m² hr. for the lower vacuum and at thermal stress <5,000 kcal./m². hr. for the higher vacuum. (5) The effect of air on the coefficient of heat emission for a high vacuum is stronger than for a lesser vacuum. (6) The experimental value for the coefficient of heat emission (α) for a moving steam and air mixture ($Y_m W_m^2 > 2$) in a high vacuum (absolute pressure 30 mm.) is

$$\alpha = A\,(Y_m\,W_m^2)^{0.16}\,1\,\frac{-7\,PA}{PM}\,H^{-0.2}$$

and for a lesser vacuum (absolute pressure >70 mm.) is

$$\alpha = B\,(Y_m\,W_m^2)^{0.16}\left(\frac{PA}{PM}\right)^{0.14}H^{-0.2}$$

when H = thermal stress of condensation surface (kcal./m.² hr.) and A and B are functions of $\left[\frac{Ge}{Jc_{lc}}\right]$ in which Ge is the quantity of condensate formed in all tubes of a given vertical row and Jc_{lc} is the quantity of condensate in the lowest tube of that row. (7) It was also shown that these equations cover the process of condensation of pure steam. (8) For small thermal diffusion resistances the coefficient of heat emission in the lower tubes is greater than in the upper tubes. With higher thermal diffusion resistance this effect is diminished and becomes negative.

Article by V. A. Rachko *Soviet Phys. Techn. Phys.* **1**, 1957 579-593

140/I

The Triple Points of Carbon Dioxide and of Argon as Fixed Points for the Calibration of Thermometers

Holland. The International Temperature Scale for platinum resistance theremometers is defined using boiling points and freezing points as fixed points. This has the disadvantage that the pressure must be determined accurately. Also the I.T.S. between 0°C and −183°C employs no fixed points within the range itself. There are indications that discrepancies of the order of 0.04°C occur between the thermodynamic scale and the I.T.S. It is therefore proposed to use the triple-points of argon and CO_2 as fixed points since they are non-variant and are also convenient for the temperature range below 0°C. The apparatus used, consists of a copper block suspended in a vacuum by a German-silver tube. The thermometer is contained in a central hole in the block and is surrounded by eight holes which can be filled with argon or CO_2. The filling pressure of gas at room temperature is chosen so that when the temperature is just below the triple-point the holes are almost filled with solid. When using argon the whole system is cooled in liquid nitrogen while the vacuum jacket contains helium as exchange gas. When the temperature is well below the triple point, the jacket is highly evacuated to break thermal contact between the block and the liquid nitrogen. The temperature rises slowly and the corresponding resistance changes of the thermometer are followed on a Diesel horst Compensator. At the triple point the resistance remains almost constant for some time before rising again. Resistance *versus* time is plotted and the triple point taken as the point of intersection of the lines representing the initial temperature rise and the almost constant condition. The purity of the gas is of great importance: using 'spectroscopically pure' argon supplied by Philips of Eindhoven the temperature rise in 70 minutes at the triple point was 0.01°C.

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Argon supplied by B.O.C. London, containing less than 10 p.p.m. of impurity gave a temperature rise of less than 0.005°C in 75 minutes. Similarly with CO_2 (here the apparatus was cooled using dry ice and alcohol) the first gas used produced a rise of 0.003°C per hour at the triple point. An increase of the purity reduced the rate to 0.001°C/hour. The results obtained using the purest gases were:

Triple point of argon	−189.341°C
Triple point of CO_2	− 56.573°C

The reproducibility was of the order of 0.001°C.

Article by A. Michels, T. Wassenaar, T. Sluyters & W. de Graaff *Physica* **23**, 1957 89-94

Mass Transfer at Low Pressures

141/I

United States. The mass transfer from solids to a gas at low pressure is of interest in freeze drying, vacuum evaporation and other fields. This investigation is concerned with the evaporation of spheres of naphthalene into air, helium, carbon dioxide and Freon −12, at pressures from 0.1 to 3,000 μ Hg and at Reynolds numbers from 0 to 1.37. The rates of evaporation were determined by weighing. The evaporation coefficient was found to be independent of pressure over the above range, but to be higher in helium and Freon–12 than in air or carbon dioxide (about 0.08 as compared with about 0.05). Results obtained at Reynolds numbers up to 1.37 indicate that the evaporation coefficient increases with Reynolds number; thus, for naphthalene in air it increases from 0.0561 to 0.0960, as the Reynolds number increases from 0 to 1.37. This is taken to indicate that the flow of gas assists the transfer of molecules across the plane of the crystal, the resistance to which is relatively large in the case of the flat naphthalene molecules. The data support the concept of additivity of surface evaporation and diffusion resistances, being capable of prediction on the basis of the available correlations for evaporation from small spheres together with experimental values of evaporation coefficients. The results agree in a semi-quantitative fashion with the general theory of sublimation developed by Stranki and his co-workers.

Article by T. K. Sherwood & N. E. Cooke *A.I.Ch.E. J.* **3**, March 1957 37-42

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A Radioactive Ionisation Gauge and its Application to the Measurement of Latent Heat of Vaporisation

142/I

See Abstract No.: 48/II

Some Properties of Thin Metallic Films

143/I

1. Introductory Remarks

France. After a brief review of the problems relating to the interconnection between the physical properties of thin films and their structure, the author discusses the application of the electron microscope to these studies. The conclusion arrived at, however, shows that the optical properties of thin films provide a better measure and guide for the study of their structure. The refractive index and the coefficient of extinction are considered to be of prime importance. These are measured by counting the multiple reflections of a perpendicularly incident monochromatic light beam which passes first through the glass or quartz substrate. Thus the amplitude of reflection is obtained from Maxwell's equation which yields the coefficient of reflection and the phase shift. The author's results relate particularly to silver and were published in *J. Phys. Radium*, **17**, April, 1956; they show considerable differences with previously published figures.

Letter by M. Perrot *J. Phys. Radium* **18**, April 1957 280-281

Simplified Treatment of Ellipsometry

144/I

United States. A straightforward treatment of the properties of elliptically polarised light reflected from thin films has been made using only the laws of elementary optics. The results are shown to be in satisfactory agreement with experiment for films of thickness up to 1,600 Å, far beyond the region of validity of the Drude approximation for very thin films.

(*Authors*)

Article by J. A. Faucher G. M. McManus & H. J. Trurnit *J. Opt. Soc. Amer.* **48**, Jan. 1958 51-54

On the Demonstration of the Bi-Refraction of Thin Calcium Fluoride Layers Obtained by Thermal Evaporation

145/I

France. The importance of precise experimental study of the optical properties of thin transparent layers obtained by thermal evaporation has already been shown; previous experiments have demonstrated that they cannot be considered as merely homogeneous and continuous layers with parallel faces as is often done. For this study, the optical properties in the zone of total reflection are most important. The present article describes experimental analyses of interference fringes in polarised light on layers of calcium fluoride of thicknesses varying from 1/10 to 10 wavelengths, approximately. The experimental curves are compared with calculated curves obtained from the optical constants of the layers. This comparison shows that for optical thicknesses not greater than one or two wavelengths there is good accord. This disappears and the disagreement increases as the thickness of the layers increases. This seems to show that knowledge of the structure of thin layers of calcium fluoride is still incomplete. An anisotropy of these layers seems to be the only explanation that fits the facts. On one layer particularly studied, the discrepancy between the theoretical and experimental curves can be shown to be completely explicable by a slight anisotropy of the corresponding layer, with a difference of indices of 2/1,000. These results make it possible to state that the calcium fluoride layers that have been studied present a bi-refraction. This is very slight, which explains why it has nearly always no measurable effects and is masked by other phenomena.

Article by P. Bosquet & Y. Delcourt *J. Phys. Radium* **18**, July 1957

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146/I

Dispersion of the Phase Change for Dielectric Multilayers. Application to the Interference Filter

United States. Dielectric multilayers designed for high reflectance produce a phase shift which varies much more rapidly with wavelength than does that for metals. This phenomenon is important in the measurement of wavelength by the Fabry-Perot interferometer when the phase corrections become inconveniently large even if the wavelength range is restricted to that for high first-order reflection. It also influences the width of the pass-band of an interference filter. This paper describes the use of a Fabry-Perot interferometer having quartz plates coated with 9-layer dielectric films to study the dispersion of the phase change that occurs on reflection. Using the secondary wavelength standards from an iron ore, it is shown that the method of exact fractions can be employed to establish the correct numbering of the orders, but that it must be combined with a theoretical calculation of the phase change. The dispersion of phase change for a 15-layer broad-band multilayer is found both theoretically and experimentally (by the use of Fizeau fringes) to be as much as 17 times as great as for the conventional multilayers. In consequence, an interference filter incorporating two such multilayer films has some unusual properties. Chief among these are the existence of several very narrow pass-bands, and an insensitivity to variations of the spacer thickness. Experimental results are given for a 31-layer filter.

Article by P. W. Baumeister & F. A. Jenkins *J. Opt. Soc. Amer.* 47, January 1957 57-61

147/I

Applications of the Interference of Light in Thin Films

Holland. An account of the interference effects produced by the reflection of light from the boundaries of a thin film, is given. It is shown how the Fresnel reflection coefficients are derived from Maxwell's equations of electromagnetic propagation of light. The conditions for maximum and minimum reflectance are discussed for the simple case of two ray interference, and for the general case assuming multiple reflections inside the film. Methods of producing thin films by evaporation in vacuum are given, and the text is illustrated by diagrams of an evaporation source and vacuum chamber, and a photograph of a typical plant. The applications of films produced by these methods are discussed, and by use of the expressions derived in the first part of the paper, it is shown that the reflection from a glass surface coated with a quarter wave-length layer of a lower refractive index material ($Mg F_2$) may be reduced to 0.25%. When a quarter wavelength of material of higher refractive index (TiO_2) is deposited on the glass, the reflection can be increased to as much as 43% with negligible absorption. Combinations of layers can be produced, consisting of alternate layers of high and low index materials, to enhance the interference effects. Mirrors can be prepared which transmit one region of the visible spectrum and reflect another with very little absorption. The applications of these mirrors to colour television are discussed.

Article by P. M. van Alphen *Philips Tech. Rev.* 19, August 1957 59-67

148/I

Interferometry for the Far Infra-Red

United States. This paper describes the current effort aimed to apply new interferometric techniques to far infra-red spectroscopy out to millimetre waves, and describes the beginnings of plans to use interferometry in a telescope above atmospheric absorption.

(*Author*)

Article by J. D. Strong *J. Opt. Soc. Amer.* 47, May 1957 354-357 7

149/I

Interference Filters for the Infra-Red

United States. The present methods of isolating particular spectral regions of the infra-red are far from satisfactory. Filters composed of various materials in combination succeed in passing only certain wavelengths, but these depend on the positions of absorption bands, etc., of the materials available. An interference filter method is now described which enables any wavelength in the range 5–20μ to be transmitted, providing secondary transmission peaks are eliminated by an auxiliary filter. The filter is made up of quarter-wavelength thickness films of tellurium spaced by half-wave layers of NaCl. Both materials are deposited by vacuum evaporation, tellurium being chosen as a high-index material which may be deposited in thick layers which do not peel or crack. NaCl, a low-index material, may also be satisfactorily deposited provided synthetic crystals are used, and thickness control is achieved by evaporation of weighed samples. Second-order peaks have been shifted to lower wavelengths by using Te films thinner than quarter-wave, and an increased thickness of spacer produces narrower transmission peaks. A number of transmission curves are reproduced. The filters are capable of being coated to liquid air temperature without damage.

Article by R. G. Greenler *J. Opt. Soc. Amer.* 47, February 1957 130-131

150/I

Interference Fringes with Cylindrically Curved Thin Films

Egypt. The theory of formation and localisation of multiple beam interference fringes by a cylindrically curved interferometer enclosing a refracting medium in transmission and at reflection is given. The effect of varying the refractive index of enclosed medium on localisation is considered. The resulting interference system formed by cylindrically curved doubly silvered phlogopite mica sheets in transmission is obtained and discussed. Its formation and localisation are in accordance with theory.

(*Authors*)

Article by N. Barakat & H. Nooh *J. Opt. Soc. Amer.* 48, Feb. 1958 92-94

151/I

Nondestructive Interferometric Thickness Measurement of Thin Transparent Films

(Reference only)

Letter by R. L. Sauer *J. Opt. Soc. Amer.* 48, April 1958 275

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A Narrow-Band Optical Filter to Isolate the 4,245 Å Spectral Region
(Reference only)

152/I

Note by R. P. Thorne & R. F. Warren *J. Sci. Instrum.* **35**, May 1958 186

Multiple-Beam Fringes of Equal Chromatic Order VIII. Method of Coincidence

153/I

United States. An interferometric method of coincidence for the measurement of a large step height $\triangle t$ is reported. The method is based upon the more rigorous interference equation $n = 2tv + f$. The term f is a function of v and is necessary to account for phase changes upon reflection at the boundaries of the interference film when other than zero or π.

(*Author*)

Article by W. F. Kohler *J. Opt. Soc. Amer.* **48**, Jan. 1958 55-57

Calculator for Thin Film Measurements

154/I

United States. An attachment is described which may be fitted to the calibrated drum of a spectrometer in order to simplify the calculations involved in the determination of film thickness by observation of interference fringes of equal chromatic order. In this method of measuring the thickness of a thin film, $\triangle t$, the fringe system is viewed in the eyepiece of the spectrometer, and wavelength readings are taken of $\triangle \lambda$, the displacement corresponding to a step in the film, and of λ_1 and λ_2, corresponding to two fringes in chromatic order. The film thickness is then determined from the relation

$$\triangle t = \triangle \lambda \, n/2$$
$$\text{where } n = \lambda 2/(\lambda_1 - \lambda_2)$$

It is therefore normally necessary to calculate the order of the fringe n upon which the measurement $\triangle \lambda$ is made. In the calculator described, an engraved disc is fitted to the calibrated drum of the spectrometer, enabling the order n to be read directly, and also the film thickness in terms of Angstroem units. A diagram of the calculator disc is given showing that film thickness σ from 24 to 616 Å may be measured on the particular disc described.

Article by W. A. Feibelman *J. Opt. Soc. Amer.* **46**, Nov. 1956 994-995

The Measurement of the Thickness of Thin Carbon Films

155/I

United Kingdom. A new method for measuring the thickness of thin carbon films avoids the need for coating the surface with evaporated silver and the preparation of an abrupt edge on the film. It can equally well be used for surveying thickness variations over a considerable area, or for measuring the thickness of a tiny fragment of film. The method is most effective with very thin films (less than 20Å), *i.e.* those most difficult to measure by other methods. It is non-destructive and allows the film to be used again. A densitometer, as used for the measurement of photographic plate densities, is used to measure the light absorption of the film. Light from an intense lamp passes, *via* a mirror system, through the film to a photomultiplier (Mazda type 27M1 with maximum output of 250 μA. equal to the full-scale deflection of the micro-ammeter). The relationship between optical density obtained in this way and film thickness, as measured by standard methods, is very nearly linear (standard deviation of many results from those predicted from the curve was only 2%) Thus by standardising the output of the photomultiplier when light is transmitted through the clear glass of the slide on which the film is mounted, the meter can be calibrated to read film thickness directly. The method could be extended to measure the density of a carbon film as evaporation proceeded by arranging the mirror system inside of the evaporation plant and preliminary tests indicate that it might be applied to other thin evaporated films which show optical absorption.

Article by A. W. Agar *Brit. J. Appl. Phys.* **8**, January 1957 35-36

Two-Colour Monitoring Method for Reproducing Thin-Film Thicknesses

156/I

United States. The reflectance curve of a thin dielectric film of given refractive index can be specified uniquely within a limited thickness range by the ratio of reflections at two different wavelengths. The method of thickness measurement described uses a single photocell with alternate sampling of two colours. The light source has a rotating filter system which alternately places one of two filters in the monitoring beam. A frequency of a few cycles per second is used, and when the system is unbalanced, this oscillation is apparent in the photocell output. When the spectral intensities of the components are equal, the frequency of the output is zero. The system is compensated by providing a red-blue double dichroic polariser and a separate linear polariser. Rotation of one with respect to the other introduces a shift in the spectral transmission. This compensator allows the ratio of red to blue sensitivity of the monitor system to be set to a predetermined value. In operation, zero oscillation is obtained from a standard film by adjusting the compensator. The standard film is then replaced by a substrate and deposition commenced. As the deposition proceeds, zero oscillation will again be obtained when the reflection coefficients reach the ratio set by the initial conditions. This method will not determine the order of film thickness, which must be assessed by visual observation or by trial and error. Sensitivity in certain bands is limited by the response of the photocell, but in a typical experiment film thickness was reproducible to within a small fraction of one per cent. The two colour method has the advantage of greater sensitivity over the monochromatic method, and avoids the maxima and minima of the single colour system, when the change of reflection with thickness is momentarily zero.

Letter by A. C. Traub *J. Opt. Soc. Amer.* **46**, Nov. 1956 999-1000

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The Effect of Incident Atomic Velocity on the Structure of Evaporated Silver Films

United Kingdom. Aziz and Scott have recently shown that aggregation in vacuum-evaporated silver films may be reduced by either reflecting the vaporised silver atoms from a Teflon surface or allowing them to diffuse through nitrogen at 3 μ, before deposition. These effects have been thought to be due to lowering the energy of the metal atoms to that of room temperature, thus reducing surface mobility. However no evidence of the dependence of film properties upon atomic velocity has hitherto been produced. Examination of heat energy data in the Smithsonian Physical Tables shows that the energy due to translational velocity of silver atoms at 1,000°C amounts to only 0.12 of the total heat energy. Thus reduction of this amount could hardly influence significantly the energy liberated on condensation. A number of factors would influence the results obtained by Aziz and Scott, *e.g.* the deposited film could be shielded from heat radiation, and impurities in the nitrogen gas could give rise to oxide formation, with known marked effect upon metal volatility.

Letter by L. Holland *Canad. J. Phys.* **35**, May 1957 697

158/I

The Epitaxial Growth and Oxidation of Nickel, Cobalt and Iron on Rocksalt

United Kingdom. The structure and orientation of films of iron, nickel and cobalt and their oxides have been examined by electron diffraction. To avoid atmospheric contamination, films of the metals were prepared within the specimen chamber of the electron diffraction camera. Metal was evaporated from a helix of tungsten. A shutter was interposed between source and target during the first stages of evaporation to prevent surface contamination of the source from reaching the target and precautions were taken to avoid deflection of the electron beam by the magnetic field of the heating element. From previous work with tungsten films it is not thought that the tungsten content of the films is sufficient to influence the structures of the metal films. A specimen tilting mechanism was incorporated to simplify examination of reflection patterns. Film thickness was estimated from the transmittance of the films to visible light. It was found that a certain minimum temperature was necessary for epitaxial growth to occur suggesting that some degree of mobility in the atoms of the deposit is required in order that they may take up positions in an ordered array. For metals on rocksalt there is the further possibility that orientation may result from the existence of ionised atoms of the deposit metal. The oxide films examined were those occurring spontaneously at 400–500°C. under a pressure of 10^{-4} mm. Hg and also at room temperature under atmospheric pressure. The oxide film thicknesses were generally less than about 100 Å. In such films it has been suggested that oxidation probably occurs by diffusion of metal ions through the lattice. It is concluded that when epitaxial growth occurs on a single-crystal substrate, either by deposition from the vapour or by oxidation, the orientation mainly observed is governed chiefly by a tendency to continue the substrate structure across the boundary. However, if the lattice misfit is very large (20–30%) incomplete orientation may occur or epitaxial growth may persist for only a small number of molecular layers. There is no indication that twinning and rotational slip are essential to the formation of an oriented overgrowth although they do serve to release growth strains in the deposit. There is some evidence, from the oxidation results, for the formation of a strained lattice where the misfit is not large as suggested in previous theoretical work. There are however many instances of epitaxy occurring when there is a larger misfit than that which would be expected to give a strained lattice. If a strained monolayer forms on the substrate surface and if the normal lattice spacing is attained in a very small number of molecular layers, the strained region may not be detected by the electron diffraction method. From the nature of the oxidation process, in which an initially complete metal lattice is changed to oxide by diffusion of atoms, it seems plausible that strained regions may form to a greater extent than would be expected in a film forming by deposition from the vapour.

Article by L. E. Collins & O. S. Heavens *Proc. Phys. Soc.* **70B**, March 1957 265-281

159/I

Optical Properties and Oxidation of Evaporated Titanium Films

See Abstract No.: 11/III

160/I

Some Properties of Thin Metallic Films. II. Study of Thin Metallic Gallium Films

France. The author's work is concerned with the variation of the coefficient of reflection as a function of the thickness of the gallium films. The film reflectivity was measured during the evaporation process for three wavelengths: 4,358, 5,461 and 5,780 Å. The rate of deposition of the Ga was of the order of 10 mμ/h. at 4×10^{-4} mm. Hg. The curves show an almost constant reflectivity of 4% for thicknesses up to 2 mμ with subsequent strongly increasing reflectivity up to 20% for thicknesses up to 7 mμ. The measurements at 5,461 Å were extended up to 50 mμ and showed the above mentioned increase continuing up to 60% at 15 mμ with a subsequent decrease to 45% at 40 mμ and a further slight increase to 50% at 50 mμ. It was also found that slow ageing in air changes the reflectivity by about 20% in an irreversible manner.

Letter by J Tortosa *J. Phys. Radium* **18**, April 1957 281-282

161/I

Influence of Purity, Substrate Temperature, and Ageing Conditions on the Extreme Ultra-Violet Reflectance of Evaporated Aluminium

United States. Aluminium coatings have a higher reflectance than other metals in the extreme ultra-violet but the reflectance of evaporated aluminium films is much more dependent on the purity of the metal and the evaporation and ageing conditions than it is at longer wavelengths. This paper reports on the effects of purity of the aluminium, substrate temperature during evaporation and ageing conditions; the effects of speed of evaporation, pressure and film thickness have been reported in a previous paper (*J. Opt. Soc. Amer.*, **46**, 1956, 1009). The wavelength range covered was 900–2,200 Å. It was found that to obtain the highest reflection it

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was necessary to use aluminium of the highest purity on an unheated substrate. Thus, the reflectance of 99.99% aluminium at 1,700 Å was 80%, measured at 6° incidence, whereas that of 99.5% aluminium (impurities iron plus smaller quantities of copper and silicon) was 70%. Similarly, if a substrate temperature during evaporation of 200°C was used instead of 50°C, the reflectance was reduced from 91% to 81% at 2,200 Å, and from 18% to 15% at 900 Å. The effect on reflectance of an oxide layer was determined theoretically from the optical constants of aluminium oxide and aluminium, which were measured experimentally. The calculated curves of reflectance against oxide thickness show that the oxide layer has little effect at 2,200 Å but at 1,216 Å and 900 Å the reflectance decreases rapidly with increasing oxide thickness and becomes only a few per cent at a thickness of 50 Å (normal incidence). Subsequently the curves pass through a series of maxima and minima, eventually reaching a value of about 12% when the surface is covered with a thick opaque layer of Al_2O_3. It is pointed out that, if oxidation cannot be prevented, it may be advantageous, for wavelengths below about 1,100 Å, to use on opaque coating of aluminium anodised to produce a surface coating of Al_2O_3 of optimum thickness. Further results showed that the growth of the oxide could be retarded by storing the coatings in a desiccator and that intense ultra-violet radiation should be avoided as much as possible, especially if water vapour is present.

Article by G Hass W. R. Hunter & R. Tousey *J Opt. Soc. Amer.* **47**, December 1957 1070-1073

Reflectance of Evaporated Aluminium in the Vacuum Ultra-Violet

162/I

United States. Al is the metal most used for coating mirrors for use in the ultra-violet, and the article describes an investigation into the way the reflectance of Al varies with conditions of deposition and age, in the far ultra-violet. The evaporation plant used consisted of a 17-inch. glass bell-jar mounted on top of a stainless steel tank, the pumping tube leading out of the side of the tank and passing *via* a right-angled baffle to the diffusion pump. The evaporation source, consisting of six helical filaments of 62 mil. tungsten, was supported near the base of the tank on Cu leads capable of carrying 1,000 A to allow fast evaporation. The glass plates were mounted in the bell-jar about 20 inch. above the source, and were cleaned by a glow discharge of up to 90 mA and 5 kV, for about 5 min. in air at a pressure between 10^{-3} and 10^{-2} mm. Hg. The material used was 99.99% pure Al and, during the warm-up of the source, the glass was shielded by a leaf-type shutter. It was possible to deposit a uniform, opaque film in less than 1 sec. To make reflectance measurements, the mirrors were placed in a cylindrical chamber receiving radiation from the exit of a vacuum monochromator. A glass plate coated with a phosphor was used in conjunction with a photomultiplier as detector, and for rapid measurements at angles of incidence between 6° and 86°, both mirror and detector could be turned simultaneously by a small motor, the detector turning through twice the angle of the mirror. The output from the photomultiplier was recorded on a Brown recorder. As a check against polarisation of the radiation, the whole apparatus could be rotated through 90°. Measurements were made in the range 800–2,000 Å, and show that by changing the evaporation time for a film 800 Å thick from 55 sec. at 10^{-5} mm. to about 2 sec., the reflectance may be increased by more than 10%. Films made at 10^{-4} mm. were generally worse than those made at 10^{-5}, though the difference was small when the evaporation was carried out in 2 sec. It is concluded that poor results are due to oxygen being trapped in the film, so that establishing full evaporation rate before opening the shutter and working rapidly and at low pressure gives the best results. The effect of age was studied up to about 20,000 hours. Little change in reflectance was noticed for wavelengths above 2,000 Å, but at 900 Å reflectance fell steadily from 20% to below 5%. Measurements at different angles of incidence showed a continuous increase from normal to grazing incidence.

Article by G. Hass W. R. Hunter & R. Tousey **46**, December 1956 *J. Opt. Soc. Amer.* 1009-1012

Preparation and Photoconductive Properties of Cadmium Telluride Films

163/I

See Abstract No.: 35/III

Photoconductivity Speed of Response for High Intensity Excitation in Cadmium Sulphide and Selenide

164/I

United States. It is possible, by measuring the speed of response in cadmium sulphide and cadmium selenide, to estimate (*a*) the density of hole traps and electron traps, (*b*) the free-electron-free-hole cross-section and (*c*) the product of capture cross-section and density of empty recombination centres in the sulphide. When the density of photoexcited free electrons in an *n* type photoconductor exceeds the density of photo-excited trapped electrons, the measured decay time of the photocurrent is equal to the lifetime of a free electron and is related to the gain and to the mobility of the free electrons according to the equation:

$$\text{Gain} = \frac{\text{Measured decay time} \times \text{Mobility of a free electron} \times \text{Applied voltage}}{\text{Square of distance between electrodes}}$$

where the gain is the number of electrons passing through the crystal between the electrodes per unit time for one photon absorbed per unit time. To obtain the conditions in which the density of free electrons exceeds the density of trapped electrons for CdS and CdSe, it is necessary to use a high illumination level. When the density of free electrons is less than the density of trapped electrons, the measured decay time will be larger than the lifetime of a free electron. If the decay time is then substituted in the equation above, the gain calculated will be larger than the observed gain. From the value of the free electron density at which the measured decay time becomes equal to the true electron lifetime, the density of trapping centres may be estimated. The author considers a model made up of three levels (1) trapping levels near a conduction band, (2) recombination levels near the centre with large capture cross section for electrons, (3) recombination levels in the lower half with small electron capture cross sections once a hole has been captured. Increase in density of the third type results in increased photosensitivity. Most recombination will however occur in level (2) and this will determine the lifetime of a free electron. Estimation of the density of recombination centres

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can be made from the values of the free electron density. Single crystals of 'pure' CdS and 'pure' CdSe as well as sintered crystals containing Cl and Cu and evaporated layers of CdSe:Cl:Cu were tested. Contacts were made using indium electrodes or silver paste and the illumination was chopped using a rotating wheel with a 3° segment removed. The growth and decay times were shown on an oscilloscope and the quantities measured were (*a*) the growth time after a long dark period and (*b*) the decay time after a short light pulse. The rise time was determined by finding the speed of the wheel at which the photocurrent rises to half the ultimate for steady illumination. The decay time was taken as that required for decrease to half amplitude after a pulse of 100 μ sec. The distance between electrodes was varied between 1.2 mm and 0.3 mm and the photocurrent measured for incident radiation of 174° ft. c. at 5,000 Å wavelength from a source at 1,900°K. The minimum response times are calculated to be 250 μ sec rise and 300 μ sec decay for CdS and 17 μ sec rise and 8 μ sec decay for CdSe. The density of traps in CdS samples was calculated to be 3×10^{14} per cm.^{3}, that in CdSe about 10^{13} per cm^3 in insensitive samples, increasing to 10^{15} per cm^3 in sensitive samples.

Article by R. H. Bube *J. Appl. Phys.* **27**, Oct. 1956 1237-1242

165/I

A Method for Deriving the Acoustic Absorption Coefficient of Gases from Measurement of the Decay-Time of a Resonator

United Kingdom. Describes a method giving direct measurement of small values of absorption coefficient, based on the well-known method of measuring reverberation time. In the experiments described, the gas is contained in a Pyrex cylinder 60.1 cm. long and 9.80 $\pm$ 0.01 cm. diameter and is excited intermittently at one of the eigenfrequencies of the tube. The amplitude of the sound is recorded as it decays after a burst of excitation and from the time-constant of the decay the absorption coefficient is calculated. The absorption due to 'classical' processes is calculated from kinetic theory, when additional absorption, due to molecular relaxation, can be estimated. The output from an r.f. oscillator is interrupted to provide the 'tone-bursts' and is then fed *via* an amplifier to a ribbon loudspeaker constructed from an Al-ribbon stretched across a length of rectangular waveguide. The loudspeaker is coupled by a horn to a probe tube of 0.042 inch i.d. and 0.058 inch o.d. to one end of the resonance tube, while a condenser microphone is similarly coupled to the other end. The whole assembly of tube and transducers is made gas-tight by means of O-rings and joints are cemented with Araldite. The system can be evacuated by a mercury diffusion pump prior to filling with the test gas. The temperature is controlled by a water-bath to within 0.1°C. It was found possible to make measurements in the frequency range 1–25 kc/s and over a range of values of frequency/pressure of 1–45 kc/s atm^{-1}. Decay times from 20m/sec to 1.5 sec were recorded, the shorter on a double-beam c.r.o. and the longer on a logarithmic pen recorder. A great advantage of the method is that the loudspeaker is silent whilst the decay curve is being recorded, so that transmission through the solid parts of the apparatus, and electrical pick-up, are avoided. Equations are presented for the frequencies of various longitudinal and radial modes of resonance, of which a considerable number were excited in the experiments, and for calculating the power losses due to shear viscosity and thermal conduction in the body of the gas and also at the walls of the tube. In the experiments, with a given eigenfrequency excited, the decay time was measured and the gas pressure then varied between about 0.1 and 1.0 atm. Graphs of absorption coefficient multiplied by the ratio p/f^2 (p = pressure, f = frequency) are presented for Ar (used as a check, since there are no molecular relaxations), N_2 and cyclopropane. A value is then obtained for the relaxation absorption coefficient, multiplied by p/f^2, for cyclopropane, of 151×10^{-13} N atm sec^2 cm^{-1} with an estimated error of $\pm 5\%$.

Article by P. D. Edmonds & J. Lamb *Proc. Phys. Soc.* **71**, January 1958 17-32

16 — CHEMISTRY — 16

166/I

Vapour Pressure of Plastic Materials

See Abstract No.: 107/II

167/I

Microchemical Analysis

United Kingdom. The technique and apparatus used in the various types of microanalysis are described and methods available for the microanalysis of ferrous and non-ferrous alloys are critically reviewed. The term 'microanalysis' applies when the analysis is completed on only a few milligrammes of sample and is taken to include both complete analysis and the determination of a single constituent. Methods of analysis by microvolumetric and microgravimetric means, absorptiometry and polarography are outlined. Electroanalysis may be by electrogravimetric or coulometric methods. Electrometric methods described include potentiometric and amperometric titrations. Radiochemical, chromatographic and ion-exchange methods are also reviewed and the microanalysis of gases is outlined. The application of microanalysis in determining the elements present in iron and steel, aluminium alloys, copper alloys, tin and tin-base alloys, lead-base alloys and zinc-base alloys is then explained. The determination of traces of gaseous elements in steel can be carried out by a method developed by Gregory. A small steel sample is heated to a high temperature in a graphite crucible in an evacuated system by a high-frequency heater. Any oxygen in the sample is converted to carbon monoxide by reaction with the crucible and this gas, together with hydrogen and nitrogen, is rapidly removed from the furnace by a mercury diffusion pump. The total gas from the sample is collected in the apparatus and analysed *in situ* for the various constituents by a fractional-condensation method combined with oxidation at a platinum filament. With this method gaseous elements at the 10–100 p.p.m. level in steels can be determined with an accuracy better than ± 10 p.p.m. using samples of 50–200 mg. The samples are heated at

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1,700°C, and at this temperature gas evolution is complete in about 5 min. The vacuum-fusion method has been criticised as being slow and complicated and a process has been developed by Smiley which permits the rapid analysis of oxygen without the use of a high vacuum. The small sample is dropped into molten platinum in a graphite crucible and any oxygen in the sample reacts to form carbon monoxide which is swept out by a stream of argon at atmospheric pressure. A modified form of Schütze's reagent is used to oxidise the carbon monoxide to carbon dioxide which is condensed in a capillary trap and measured with a capillary manometer. Each determination takes about 12 min. In a similar technique for measuring small amounts of hydrogen the sample is burned in a stream of purified oxygen at about 1,000°C and the water produced is collected in a capillary trap at −78°C. The hydrogen content is finally calculated from vapour-pressure measurements at 100°C.

Article by G. W. C. Milner & J. W. Edwards *Metallurgical Review* **2**, No. 6, 1957 109-155

Purification of Chemicals by Zone Melting 168/I

United Kingdom. The development of semi-conductor devices such as the transistor called for the production of germanium and silicon of exceptionally high purity. The technique of zone melting which was originally devised by Pfann to meet this demand has been extended to the purification of a number of inorganic and organic chemicals. It is a well-known metallurgical phenomenon that in the course of cooling of a liquid metal solidification starts at the bottom. A solid front of metal, in a purer state than the original, advances behind the diminishing liquid portion which retains the impurities in solution. Thus the impurities previously dispersed throughout the metal will mostly be concentrated in its top layer when solidification has taken place. The zone melting purification process consists of heating above its melting point a bar or tube of the substance to be purified, and moving the molten region along the substance so that recrystallisation in a pure state takes place behind the zone, leaving the impurities concentrated at one end of the bar. Typical organic substances which have been purified in this way are Acetanilide, Pyrene, Anthracene, Chrysene and Halowax. Benzoic acid of 99.91 mol. % purity gave a yield of 40% of 99.997 mol % purity after three zonal purifications, 85% of the impurity present being removed each time. Eleven recrystallisations from benzene or 25 from water are necessary to produce benzoic acid of the same purity. The process has four main advantages: Being continuous it is fast, the impurities are automatically separated, handling of the chemicals is minimised and recovery of the starting materials complete. The article gives a detailed description of an apparatus capable of dealing with 0.15 to 5 g. of material and of another treating amounts of 1 kg. While metallurgical zone melting is performed horizontally, organic chemicals require a vertical tube. In the smaller equipment the heating zone is obtained by exposure to the radiant energy of an incandescent lamp, in the latter a ring furnace is used. This furnace is moved down a four foot tube of material at 1.5 inch. per hour and is automatically returned to the top of the end of its travel. It takes 24 hours for a molten zone 2.5 inch. long to travel down the tube, and after seven days an anthracene impurity of 0.2% by weight in naphthalene was reduced to 0.00002% in half the tube. The slow zone speed is necessary to allow crystal formation without entrapment of liquid. In other apparatus the zone can be created by r.f. heating or, as was done for naphthalene, by lowering the tube of material through a hollow cylinder which is temperature-controlled by chambers of boiling chloroform and refluxing trichloroethylene separated by a vacuum jacket. It has been proposed to extend these techniques to the separation of heavy water from ordinary water, the treatment of photographic chemicals such as the silver halides, and to the purification of metal ingots weighing several tons.

Article by R. Handley *Mfg. Chem.* **27**, Nov. 1956 451-453

Surface Properties of Magnesium Oxide 169/I

Egypt. In a study of the surface properties of magnesium oxide, brucite was selected as the raw material for calcination to avoid the influence of certain factors if precipitated hydroxide or carbonate of magnesium is used. The adsorption of n-hexane and cyclohexane on the products was measured. When calcination was effected in air for 5 hours there was progressively increasing surface area at 350° to a maximum at 500° and then a decrease at 650°, 800°, 950° to a minimum at 1,100°. At a calcination temperature of 500° there was a decrease in surface area at 0.5, 1, 2, and 3, 5, and 5 hours of from 93–36.1 $m.^3/g$. Reduction of surface area appears to be a direct result of sintering rather than adsorption of an air film at the oxide surface. Dehydration was also studied under vacuum for 5 hours. Maximum surface area is found at 350°. Activity is little affected by rise in temperature under vacuum until 650° is reached, at which temperature the surface area diminished rapidly with increasing temperature to a very small amount at 1,100°. The effect of duration of heat treatment under vacuum on adsorption of cyclohexane by magnesium oxide was studied for various temperatures of dehydration and time intervals of 0.5–15 hours. At 950° or 1,100°, adsorption capacity falls rapidly with increase of time of heating, but there is a limiting isotherm obtained by heating brucite at 5 hours. The activity of solids, as revealed in their adsorptive capacity, has been investigated particularly in respect of the dehydration of brucite and the development of sintering. Dehydration under vacuum for 5 hours gives products of greater areas than of those obtained in presence of air.

Article by R. I. Razouk & R. S. Mikhail *J. Phys. Chem.* **61**, July 1957 886-891

Thermal Effects in the Chemisorption of Oxygen on Nickel: A Comparative Investigation Using Powders and Evaporated Films 170/I

See Abstract No.: 57/III

Radiation Quenching, Ion Bombardment and Annealing of Nickel and Platinum for Ethylene Hydrogenation 171/I

United States. An improved arrangement is described for investigating the activity of surface catalysts in the hydrogenation of ethylene. High vacuum techniques are employed to eliminate spurious and contaminating

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effects and various cleaning techniques are then applied. The reaction chamber is isolated from the rest of the system by cold traps and metal high-vacuum valves which can be out-gassed. Three-stage fractionating oil diffusion pumps of borosilicate glass, backed by mechanical oil pumps are sealed to the cold traps to avoid grease or wax joints on the reaction chamber side and an evaporated molybdenum getter is used to improve the vacuum. The catalyst, a 1 cm. square of Pt or Ni, is mounted on a magnetically operated carriage. A shutter prevents evaporated or sputtered metal from entering the compartment where the catalytic activity is determined. Initial outgassing for half an hour by induction heating is followed by one or more of the three treatments: (*i*) radiation quenching by suddenly stopping the induction heating current and allowing the catalyst to cool to room temperature in vacuum; (*ii*) bombardment by positive argon ions; (*iii*) short annealing for a few minutes at 500°C. Catalytic activity is then determined for the different combinations. Results with nickel show high catalytic activity following radiation quenching or argon ion bombardment but this activity is very much reduced by subsequent annealing, suggesting that the high activity is caused by surface lattice defects which are largely removed by the annealing. The higher activity of bombarded nickel compared with radiation-quenched nickel does not appear to be entirely due to increase in surface area. Low-energy electron diffraction observations of an ion-bombarded surface show a distorted structure which may also account for the observed greater stability of such surfaces compared with those produced by radiation-quenching. The similar comparative activities of bombarded and radiation-quenched platinum support these conclusions and stress the need for avoiding both contamination and structure effects if the characteristic catalytic activity is to be obtained from a particular solid.

Article by H. E. Farnsworth & R. F. Woodcock *Ind. Engng. Chem.* **49**, Feb. 1957 258-260

172/I

Hydrogen Sorption and the Parahydrogen Conversion on Evaporated Nickel Films

United States. The author has studied the changes which may occur in the electrical resistance of nickel films when hydrogen is absorbed or adsorbed. The work was carried out to provide information concerning hydrogen sorption on nickel surfaces at −78°C and the kinetics of the parahydrogen conversion in the presence of nickel as a catalyst. The pure nickel films were deposited by evaporation from an electrically heated filament in high vacuum on to the wall of the Pyrex glass vacuum chamber. Platinum electrodes were sealed into the wall of the chamber, and the resistance of the nickel films deposited across these was measured using a sensitive Wheatstone bridge. The vacuum system was adequately trapped and outgassed. Hydrogen pressures were measured by a mercury manometer and a Pirani gauge was used at low pressures down to 2×10^{-6}mm. Hg. During evaporation, the substrate was held at 0°C, and the film was afterwards annealed at 80°–90°C, followed by cycling between −195°C and 80°C. Annealing was assumed to be complete when the resistance at 0°C repeatedly returned to the same value. The results show that three processes can take place. Firstly there is a decrease in resistance of the film due to initial adsorption of hydrogen. This is shown in a graph, for both thick and thin films, in contact with hydrogen at −78°C and at pressures below 2×10^{-6}mm. Hg. This change in resistance was found to be irreversible by prolonged pumping. The second effect takes place at gas pressures between 10^{-6}mm. Hg and 600 mm. Hg, when rapid reversible adsorption of the gas occurred. The third effect is a slow irreversible absorption of gas, which gradually decreases the resistance and poisons the catalytic properties of the film. A comparison of the resistance changes caused by the three different effects is given in a table. In another table are given the rates of p-hydrogen conversion for eleven different nickel films. The p-hydrogen content of the reaction products was obtained by sampling the gas and making thermal conductivity measurements. Some measurement of activation energy was made on films containing absorbed hydrogen, and these results are given in a third table. In the discussion which follows reference is made to previous work concerning the annealing of thin films, and the associated resistance changes. The mechanism of hydrogen sorption is discussed and it is concluded that the previous assumption of a single sorption mechanism must be re-examined. The results obtained for the p-hydrogen conversion are compatible with the Bonhoeffer-Farkas theory. The poisoning action is inferred to be due to a reduction in the extent of surface adsorption thereby limiting the reaction.

Article by J. H. Singleton *J. Phys. Chem.* **60**, Dec. 1956 1606-1611

173/I

Chemisorption of Gases on Nickel Films I. Kinetic Studies

Article by P. M. Gundry & F. C. Tompkins *Trans. Faraday Soc.* **52**, Dec. 1956 1609-1618

United Kingdom. Lack of agreement in the interpretation of the slow rate of sorption of hydrogen and carbon monoxide by evaporated iron films and by nickel films led to a detailed investigation of the nickel + hydrogen system. This paper deals only with the kinetic aspects of the work, other results are dealt with in a subsequent report.

174/I

Ionisation Gauge Detector for Gas Chromatography

See Abstract No.: 49/II

175/I

Some Features of Gas-Liquid Chromatography

United Kingdom. Considers the process in which the components of a gaseous mixture are separated by passage along a column packed with a finely-divided solid supporting a non-volatile liquid. The mixture to be analysed is injected into a stream of inert gas which carries it into the column as vapour, and different components of the vapour emerge from the other end of the column after varying intervals. The emergence of a component is usually detected by an element similar to that of a Pirani gauge, the changes in resistance of the filament being recorded continuously. Quantities of interest are the 'retention time' of a component, which is the

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interval between injection of the mixture into the stream and the first emergence of the component, and the 'exit time' which is the time during which the component continues to emerge. A previous theory of the operation of the column assumed that diffusion mainly controls the processes, but the author suggests that it is rather a matter of the solubility of the various components in the non-volatile liquid in the column. A simplified picture imagines a localised 'closed circuit' in which vapour from the 'front' of the charge dissolves in the liquid in the column, to re-evaporate after the passage of the charge. This closed circuit moves along the column, with increasing velocity and length, and the circuits containing different components in the mixture separate as they move with different speeds. The solubility of each component is thus the critical factor, and an equation is developed to give the retention time of a component in terms of the retention time of an insoluble component, the effective diameter of the pores in the packing, the thickness of the film of liquid on the packing and the solubility of the component considered. Another equation for the exit time is obtained by considering the length and speed of the relevant 'circuit' when it reaches the end of the column. This equation also involves the solubility. The potential resolution of a column is considered to be equal to the ratio of the retention time to the exit time, and the factors restricting the resolution actually obtained are considered. It is concluded that it will often be an advantage to operate the column at reduced temperature. Other factors considered are the pressure in the column, which is lowest at the exit, and the arrangement of the hot wire used as detector. The pressure should not be so low that the flow departs from ordinary viscous flow, while the hot wire, if mounted axially, should be as short as possible. The results of some test experiments are given and discussed. These used known mixtures of paraffins with di-nonyl phthalate as the stationary liquid in the column, and a measure of agreement between measured and calculated retention and exit times is noted. In four appendices, there is some discussion of the way to measure the height and width of the peaks observed on an actual chromatogram, and how to obtain the area of a peak, which should be proportional to the amount of the particular component present in the mixture. The effect of the physical characteristics of the packing is also discussed, together with the conditions necessary to avoid turbulence.

Article by G. Burrows *Trans. Inst. Chem. Engrs.* **35**, No. 4 1957 245-257

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Composition of Lubricating Oil 176/I

See Abstract No.: 173/III

On the Decomposition of Liquid Hydrocarbons by Small Electric Arcs 177/I

Germany. The article is a shortened account of a recent lecture and describes experiments on the thermal decomposition of liquid hydrocarbons by electric arcs of short length and short duration (10 ms).

Article by H. Kroepelon, H. Kadelbach, H. Kopsch & E. Winter *Chem. Ing. Tech.* **28**, Nov. 1956 703-706

Determination of Carbon and Hydrogen in Organic Fluorine Compounds 178/I

United States. In micro-combustion determinations of carbon and hydrogen in organic fluorine compounds, the fluorine in the combustion products may react with the silicon combustion tube to form silicon tetrafluoride. This is absorbed in the combustion train and causes high results. A method is described in which fluorine is absorbed by magnesium oxide. The quartz combustion tube contains a central length of copper oxide in wire form, flanked at each end by magnesium oxide and then by rolled silver gauze. The tube is heated in a furnace maintained at 900°C. Air or oxygen for combustion is purified by passing it over copper oxide at 800°C and then through a scrubber containing ascarite to remove carbon dioxide and dihydrite to remove water. For compounds containing nitrogen, an extra scrubber containing manganese dioxide is provided. The combustion products pass onto absorbers containing, respectively, dehydrite, manganese dioxide, ascarite, and ascarite-dehydrite. The apparatus is suitable for testing solids, liquids, or gases. Solids are weighed into platinum boats, and liquids are introduced into capillaries which are placed on platinum trays. Gas samples are transferred from pressurised metal cylinders through a micro-pressure vessel to a water-jacketted gas burette filled with mercury and having a levelling tube. The height of the mercury can be regulated by a mercury lift. The burette communicates through a 4-way stopcock with the micro-pressure vessel, a vacuum pump with cold traps and manometer and a delivery tube to the combustion tube. A side branch of this conducts the oxygen into a platinum tube which injects it direct into the hot silver gauze, thereby obviating explosions or flashing. The gas sample is pushed into the combustion tube at the rate of 0.2 ml/min. by means of the mercury lift. The absorbers are removed and weighed 50 minutes after the gas was introduced into the combustion tube. Some results obtained in carbon and hydrogen determinations, using a conventional filling (metallic silver and copper oxide at 900°C and lead peroxide at 195°C), are given. Even in the first combustion they were high, and the discrepancies increased with subsequent combustions. Improvements shown when magnesium oxide was used are tabulated for fluorine-containing, and some non-fluorine containing compounds. Reasons for failure of the magnesium oxide filling to remove all the fluorine are discussed, and the differences in precision in carbon and hydrogen determinations in fluorine compounds and in non-fluorine compounds and in non-fluorinated compounds have been examined.

Article by R. N. McCoy & E. L. Bastin *Analyt. Chem.* **28**, Nov. 1956 1776-1780

Method for Extraction of Ions Formed in Rapid Combustions 179/I

Belgium. It is known that high temperature chemical reactions produce ions and this is true of flames. Several scientists have found methods of estimating the number of these and published results between 10^6 and 10^{12}

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Article by L. Vanreusel & J. M. Delfosse *Ann. Soc. Sci. Bruxelles* **70**, No. 1 1956 67-72

ions per cm.^{3}, but no study has been made of the nature of these ions. As it is generally accepted that there are about 10^{11} ions per cm.^{3}, this would correspond, for a flow of 5 cm.^{3}/sec. of gas, to an ion current of about 10^{-8}A. This can be measured in a mass spectrometer. The major difficulty lies in the fact that the pressure of the flame is necessarily about a million times greater than the pressure normally employed in a mass spectrometer. This problem is solved in the apparatus described by means of intermittent communication between the combustion chamber I and the analysis chambers II and III. Details of the arrangement are given.

180/I

The Shock Tube—Just a Piece of Pipe

Article by G. P. Wachtell *J. Franklin Inst.* **264**, Sept. 1957 241-243

United States. Briefly describes the shock tube in the Franklin Institute Laboratories, used to study phenomena in rapidly moving gases. It consists of a driver tube 22 ft. long, containing helium at high pressure which acts as a 'driver'. The driver tube is separated from the experimental section by a cellophane diaphragm, which can be punctured by an electrically-operated plunger to release the driver gas into the experimental gas. This latter is maintained at reduced pressure by a vacuum pump, and when the diaphragm is torn, a shock wave moves at supersonic speed into the experimental gas, eventually striking the far end of the experimental tube and being reflected as another shock wave. Part of the experimental tube is made of glass to observe ignition when a mixture of combustible gases is investigated. The speed of the shock wave is measured by observing the time between its passage through a light beam and its arrival at the end of the tube. There is a photograph of the equipment, but no further details.

181/I

Storage of Free Radicals at Low Temperature

Article by Anon. *Engineer* **202**, 7.12.1956 828-829

United States. Highly reactive molecular fragments, such as unstable atoms or free radicals, particularly N, O and OH, are known to exist in flames and hot gases. At the National Bureau of Standards such free radicals are produced in an h.f. discharge maintained in a wave-guide resonator at 2450 Mc/s and are then frozen very suddenly at 4.2°K on a quartz window surface in contact with a liquid helium bath. The solid deposits possess unusual properties, such as the emission of bright glows, blue 'flames' and coloured flashes of light. Spectroscopic and colorimetric analyses provide information on the structure of the radicals and molecules formed on slight heating from 4.2°. The apparatus is capable of storing the radicals for many hours and samples of nitrogen kept in the apparatus showed a green afterglow persisting for several minutes with a decay half-time of about 15 sec. Oxygen produces a clear glassy deposit with complex absorption spectra and on warming to 20°K leaves a violet deposit which is a mixture of 0 and 0_3.

182/I

The Design and Performance of Small Climbing Film Evaporators

See Abstract No.: 111/III

183/I

New Method Portends Small Tall Oil Units

Note by Anon. *Chem. Engng.* February 1957 144

United States. Tall oil and vegetable oil residues can be refined more cheaply with the use of a new technique for the anhydrous fractionation of these substances, developed by Spence & Green Chemical Co. The method uses a vacuum of 300 μ Hg as compared with about 5 mm. Hg for steam fractionation, and a lower operating temperature. A 15-ton per day unit could make rosin and fatty acid at less than three-quarters of the cost of a unit involving steam fractionation.

184/I

Improvements r.t. the Treatment of Materials by Electron Bombardment

M. C. Crowley-Milling & Metropolitan-Vickers Electrical Co. Ltd. *Brit. Pat.* 764,337

United Kingdom. Very rapid sterilisation of foods, chemicals, medical and surgical apparatus, etc. can be effected by means of high energy electron beams which are capable of penetrating to a great depth and thus of destroying living organism, such as bacteria. In the continuous sterilisation the material to be irradiated is moved across the beam which hitherto has been of approximately circular cross-section. The unequal dosage is thus concentrated at the centre of the article. This disadvantage is overcome by applying a magnetic field from a pair of flat-faced pole pieces splayed outwards to bend the beam and to spread it laterally. One design of the apparatus allows the irradiation of the underside of the material by deflecting one half of the beam through 180°. The preferred arrangement consists of a linear accelerator, van de Graaff generator or Betatron as electron beam emitter in a vacuum chamber with a metal window.

185/I

Influence of Initial Vacuum on Steam Sterilisation of Dressings

United Kingdom. It is generally accepted that for the efficient sterilisation of surgical dressings it is desirable to remove the air completely from the steriliser and its contents. Residual air delays the penetration of steam into dressings and renders the attainment of sterilising temperatures slow and unreliable. Little evidence is available on the merits of the different methods presently used to remove the air. Details are given of an investigation into the influence of different degrees of preliminary vacuum on the time taken to reach an arbitrary sterilising temperature in surgical packs which were deliberately overloaded and badly sited in the steriliser. The air was removed from a jacketed autoclave with an oil-sealed rotary type of vacuum pump protected by a condenser. Dry saturated steam was supplied at 20 p.s.i. pressure. A standard drum of hucka-

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back towels was placed in the position where the longest time was taken to reach the desired temperature in order to impose the severest test on the efficiency of the evacuation method. Times were plotted for the temperature inside the drum to reach the sterilising temperature of 115°C for values of the initial pressure ranging from 10 to 1,000 mm. Hg. With a high initial vacuum (pressures below 20 mm. Hg) the time was consistently about one minute, at pressures greater than 300 mm. Hg the points were scattered and unpredictable but all times were in excess of 30 minutes. In further experiments the steriliser was packed full with dressings and utensils, and envelopes of test bacillus were scattered about the load. With an initial pressure of 15 mm. Hg, 100% sterilisation was obtained after 10 minutes exposure to steam. It is clear that if rapid and predictable sterilisation is required in an autoclave, the pump should be capable of rapidly reducing the pressure in the chamber to a pressure of at least 20 mm. Hg.

Article by R. Knox & E. J. K. Penikett *Brit. Med. J.* March 1958 680-682

Management of an Artery-Graft Bank

186/I

United Kingdom. The use of β–propiolactone to sterilise artery grafts which are then preserved by deep-freeze refrigeration, freeze drying or storage in physiological saline solution or formalin has been investigated as a means of building up a bank of homologous grafts. Sterilisation in 1% β–propiolactone makes it possible to use grafts which are not initially sterile and which, under cold storage conditions, may be collected up to 24–36 hours after death. Only cases with specific infective diseases are avoided. Each specimen is washed in sterile physiological saline solution and stored in a deep-freeze refrigerator until it can be trimmed and described on a record card. It is then placed in a sterile tube (6 inch. × 1 inch rimless) and incubated with 1% β–propiolactone for two hours at 37°C. After this the mouth of the tube is flamed, the graft is washed in sterile salt solution, covered with sterile gauze and stored. Tubes have been stored in a deep freeze refrigerator at −70°C. Before use they are allowed to warm to room temperature. The caps are then removed and a sterile saline solution containing streptomycin and penicillin is added. Freeze drying in high vacuum makes it possible to obtain grafts of good keeping properties which can be sent anywhere. Freeze drying is carried out in two stages which remove about 98% of the water in the tissue. Most of the water is extracted in the first stage using a vacuum of 1–0.5 mm. Hg. The mouths of the tubes containing the grafts are covered with sterile gauze and two-thirds of each tube is immersed in a mixture of alcohol and solid carbon dioxide. The tubes are then transferred to the vacuum drying apparatus where they are surrounded by brine and ice at −10°C. A cold condenser, kept at −74°C by alcohol and cardice, and a phosphorus-pentoxide trap are included in the vacuum system. The pressure is measured by a Pirani gauge. When the apparatus has been assembled the joints are sealed with 'Q' compound and the system is pumped for 2–3 days. Before secondary drying, each tube is sealed with a sterile rubber cap and melted picien wax. While the wax is still soft it is swabbed with alcohol and a sterile aspirating needle is plunged through wax and cap. The tubes are connected, through stopcocks, to a drying manifold *via* the needles and modified syringe fittings. The manifold is connected to the vacuum pump *via* a P_2O_5 trap. With the stopcocks closed the system is evacuated to a pressure of 0.01 mm. Hg or better. The stopcocks are then opened and the sudden change of pressure serves to clear any blocked needles and to pull the caps on to make a good seal. Secondary drying is carried out for 2–3 days. The tubes are sealed for storage by closing the stopcocks, pulling the tube and cap off the needle and sealing with more wax. A 'Viscap' is placed over the cap. The grafts are reconstituted by removing the viscap and wax, and, after swabbing the rubber cap with alcohol, plunging through a needle which is connected to a supply of sterile saline solution containing antibiotics. At least half an hour must be allowed for the graft to reconstitute. A strain of a sporing aerobic bacillus, which was the only organism observed to resist sterilisation with β–propiolactone, was not present in the same specimen tested after 26 weeks' freeze-dried storage. Grafts have also been stored in physiological saline solution in a refrigerator at +4°C. A few short lengths of grafts have been preserved in 4% formalin without the use of β–propiolactone and stored in the dark at 4°C for a maximum of six months.

Article by A. J. H. Rains N. Crawford S. H. Sharpe J.F.D.Shrewsbury & G. J. Barson *Lancet* **211**, 20.12.1956 830-832

Lyophilised Plasma

187/I

See Abstract No.: 27/IV

Preservation of Leptospirae by Drying

188/I

See Abstract No.: 28/IV

Occurrence of a Cardio-Active Principle in Spleen

189/I

Australia. An examination has been made of extracts of spleen to determine whether any cardioactive material is present in this organ. Serial extractions of freeze-dried freshly removed ox-spleen were performed in a Soxhlet apparatus using specially purified solvents. The acetone fraction contained a cardioactive material which showed a positive inotropic effect on the isolated papillary muscle of the cat.

Letter by L. B. Cobbin & R. H. Thorp *Nature* **180**, 3.8.1951 242-243

Size and Shape of Alfalfa Mosaic Virus

190/I

United States. Three kinds of macromolecular particles can be produced by plants injected with alfalfa mosaic virus. The technique for their separation is described. Electron microscope specimens were prepared using essentially the freeze-drying method of Williams. The specimens were shadowed with uranium by evaporation.

Letter by J. B. Bancroft & D. Kaesberg *Nature* **181**, 8.3.1958 720-721

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191/I

A Replica Technique for Particle Surfaces

See Abstract No.: 76/II

192/I

Energy Transfers Between Crystals and Adsorbed Dyes I. Desensitisation

See Abstract No.: 254/I

193/I

Fluidised Bed Sublimation—a New Separating Process

Article by G. Matz *Chem. Ing. Tech.* **30**, 1958 319-329

Germany. Fluidised bed sublimation suggests itself as a purification process for temperature-sensitive substances having a vapour pressure below 0.5 mm. Hg at the working temperature. In addition, to the possibility of continuous operation, this process offers improved heat transfer rate. For the fluidised medium use is made of a fluidisable abrasion-resistant solid, together with which the non-subliming residue is carried away. The fundamentals and technical details of the process are considered and applied to the special case of adsorption sublimation in a fluidised bed and also to vacuum fluidised bed sublimation.

(*Author*)

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194/I

The Application of Vacuum in Metallurgy

See Abstract No.: 52/I

195/I

High Temperature Technology

Article by N. K. Hiester F. A. Ferguson & N. Fishman *Chem. Engng.* **64**, March 1957 237-252

United States. Typical processes carried out at temperatures above 2,500°F are described and a review is given of methods of obtaining high temperatures and of construction materials suitable for use at high temperatures. Amongst the early processes which have been discontinued, mainly on economic grounds, are the fixation of nitrogen by the electric arc and the Wisconsin process, and the Hall method of producing alumina. Techniques in present use include electrothermal, calcination, pyrometallurgical and combustion processes. High-temperature technology is based to a large extent on the combustion of fuels but the maximum temperatures available from chemical reactions are limited by the temperature range in which the molecular species dissociate. Combustion reactions which produce only carbon monoxide and nitrogen give the highest flame temperatures because of the high dissociation temperatures involved. The use of the gas turbine as a chemical reactor is restricted by the temperature limitations of current materials of construction. Work by Reaction Motors, however, indicates that temperatures of 5,300–5,700°F should be possible in a rocket-engine type of reactor. The probable upper limit of temperatures obtainable by practical chemical combustion is 9,500°F. Temperatures above 10,000°F are commercially attainable by means of the electric arc and plasma. Temperatures up to 90,000°F have been observed in flames emitted from electrodes in very high current density arcs. By introducing chemical compounds or gases into these flames, new chemical species and unusual properties have been discovered. In the electronic torch gaseous molecules are dissociated in an arc discharge. Recombination of the atoms as they leave the torch produces temperatures as high as 18,000°F in the core and gives an average gas temperature of 5,000°F. Temperatures up to 3,600°F are attainable in resistance furnaces using oxide resistors and higher temperatures would be possible with resistors of higher melting point. Tantalum carbide (m.p. 7,015°F) resistors have been used experimentally but the high vapour pressure of the material proved a disadvantage. Impulse-induced temperatures are the highest non-nuclear temperatures yet produced but they are transitory. They can be obtained by means of the ballistic piston, the shock tube and the arc discharge. With an arc-discharge method, temperatures of about 450,000°F in helium and 1,000,000°F in deuterium have been attained. Advantages of solar and arc-image furnaces are that the specimen can be exposed in any environment and can also serve as its own crucible. Temperatures in the range 5,400–6,750°F can be obtained. Pressurised water nuclear reactors are operating at about 450°F and organic-moderated reactors are likely to reach 650°F. Processing temperatures of about 3,200°F may be attained in future nuclear reactors. Amongst the construction materials of possible use at high temperatures, niobium, molybdenum, tantalum, tungsten and their alloys appear to be the most promising, but research is required on their stability under various environments. The high temperature properties may be improved by coatings, for example molybdenum coated with silicon to form a thin layer of molydbenum disilicide provides satisfactory oxidation resistance up to 3,600°F. Work is also being carried out on liquid metals contained in metals of higher melting point. Drawbacks to the use of metallic compounds at high temperatures are their relatively low ductility and poor resistance to thermal shock. Materials which may provide crucibles suitable for melting titanium are Mo_3Si, TaB, ZrB_4, UB_2, CeB_4, CeS and ThS_2. A cermet which shows promise for high-temperature work is a titanium-carbide base metal-bonded material. Other cermets under study include combinations of chromium boride and nickel, boron carbide and iron. New high-temperature techniques include the Sheer-Korman application of the high intensity arc. Oxythermal processes which use thermal energy supplied by combustion with oxygen are being developed to replace electric furnace techniques. Research is also being carried out on the production of titanium carbide from rutile or ilmenite at 3,270°F, and on vapour plating in which films of the desired metal are deposited by reducing their salts in proximity with the metal to be coated.

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Corrosion Studies in High Temperature Water by a Hydrogen Effusion Method

196/I

United States. Metals can corrode through interaction with water at high temperature and pressure. Hydrogen is liberated by the reaction and its rate of formation is a direct measure of the corrosion rate. Information about the behaviour of a number of ferrous materials is important in the design of aqueous heat exchangers for nuclear power plants. The reaction is

$$3\,Fe + H_2O - Fe_3O_4 + H_2$$

The method of corrosion measurement is based on the fact that in a sealed metal container at elevated temperature, hydrogen evolved as a result of the corrosive reaction with water in the container will permeate the walls of the container at a comparatively rapid rate. The container can be made of a section of flattened annealed steel tubing 0.25 inch o.d. and 0.020 inch thick. It is filled with fluid and the ends are pinched and spot-welded to form a sealed capsule. In use, a capsule is placed into an evacuated oven of known volume. The rate of hydrogen effusion is calculated from measurement by a Pirani gauge of the rate of pressure rise in the heated evacuated system. Results show that the role of corrosion between low carbon steel and distilled water at 600°F is rapidly inhibited by the formation of an oxide film. After about 150 days the rate of attack has been reduced to 0.000036 inch. per year. Increase in the pH of the fluid does not produce any very significant change in the corrosion behaviour. Both carbon and stainless steels exhibit high initial corrosion rates, but that of the stainless steel drops very rapidly to an extremely low value. It was found that there was an increase in the time needed to produce a protective film if the carbon steel was hydrogen and vacuum-annealed prior to capsule formation. Microscopic examination indicated that the cause lay in grain growth and carbide formation which tends to accelerate corrosion. Damage to oxide films can result from rapid cooling of the metal but the corrosion rate returns to its previous value within a comparatively short time (about 10 days in one experiment). A sudden increase in temperature from 600°F to 650°F causes a corresponding increase in the corrosion rate but this then drops at a much more rapid rate than had been the case at the lower temperature. It is possible that the temperature rise causes an alteration in the protective film so that eventually the corrosion rate at either temperature would be the same. An experiment with capsules identical in all respects except wall thickness showed that the corrosion rate measurement of low carbon steel gave similar results with wall thicknesses of 0.153 and 0.028 inch.

Article by M. C. Bloom, M. Krulfeld, W. A. Fraser & P. N. Vlannes *Corrosion* May 1957 297t-302t

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The Corrosion of Iron in High-Temperature Water I. Corrosion Rate Measurements

197/I

United States. Corrosion between iron and pure water in the temperature range 240–360°C has been studied with the object of developing a ferrous alloy capable of replacing the corrosion-resistant, high-alloy steels which are used for the water-coolant systems of power-generating nuclear reactors. In this part of the paper a new method of measuring the corrosion rate of metals in high temperature water is described. The experiments were carried out on specimens of Armco iron strip and high-purity, vacuum-cast, iron rod suspended in water in a 250 ml. autoclave. This autoclave consisted of a commercial reactor assembly with the standard top replaced by a specially machined top with a built-in valve which made it possible to fill the gas space in the autoclave to a known pressure with helium. A torque of 6–8 ft. lb. was sufficient to seat the valve so that no leakage occurred at 360°C. The autoclave was heated in an oven to the required temperature, cooled to room temperature by quenching, and tumbled end-over-end for about 30 mins. It was then attached to a vacuum system, the gas was expanded into an evacuated known volume, its pressure was measured by a mercury manometer and a sample was taken for analysis. The amount of hydrogen evolved was calculated from the pressure, volume, temperature and analysis of the gas, and was taken as a measure of the rate of corrosion. Further measurements were obtained by pumping out the gas in the autoclave and repeating the cycle. The reliability of the method was tested by generating known amounts of hydrogen in the autoclaves by the reaction of weighed amounts of magnesium with the water. Oven temperatures were measured by means of a pressure gauge connected to a water-filled autoclave in the centre of the oven. Gas analyses were carried out on a General Electric mass spectrometer which, for the type of measurements required, had a precision of 3–4%. After exposure for an hour or less to water at about 300°C the surface of the specimens showed interference colours. At ten hours a blue surface film was observed which was calculated to be about 1000°Å thick. Further exposure resulted in a shiny black film which persisted for 150–200 hours, after which the film became dull black and porous. No further change was observed up to 1,500 hours. After 500 hours exposure the dull black film was about 1μ thick. The corrosion rates observed are of the same order as the rates of oxidation of pure iron in oxygen at the same temperature. Experiments on Armco iron specimens showed that their surface finish influenced the rate of corrosion. Vacuum annealing for short periods increased their corrosion rate whereas a long anneal in pure dry hydrogen gave a considerable reduction. With high-purity vacuum-cast iron the corrosion rate was increased by both types of annealing. Measurements in saturated vapour and superheated vapour showed the same corrosion behaviour as in the liquid phase, and large increases in the pH and dissolved salt content of the water had no effect on corrosion rate. It is thought that ferrous ions formed at the metal-oxide interface diffuse through the oxide by migration into vacant lattice sites. When they reach the oxide-water interface, ferrous hydroxide is formed which decomposes into magnetite, water and hydrogen. Electrons migrate with the ferrous ions and there is a counter-current diffusion of lattice vacancies. The electrons neutralise protons at the oxide-water interface and hydrogen atoms are formed, most of which combine to molecules. The entry of hydrogen into the metal lattice means that either hydrogen atoms or protons migrate from the water interface through the oxide to the metal interface.

Paper by D. L. Douglas & F. C. Zyzes *Corrosion* **13**, June 1957 19-32

Vacuum Injection Casting Furnace

198/I

United States. Description of a furnace for nuclear reactor fuel rod manufacture.

Note by Anon. *Engineer* **203**, 28.6.1957 1001

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199/I

Article by H. K. Barton & L. C. Barton *Machinery* 91, 26.7.1957 211-220

Vacuum Die Casting Processes

(Reference only)

200/I

Paper by D. Morgenstern *Modern Castings* May, 1957 80-83

Vacuum Die Casting Opens New Frontiers in Design

United States. Die casting under vacuum ensures the removal of air from the mould during pouring and thus contributes to low porosity and dense grain structure. The improved strength of the zinc and aluminium castings obtained by the method described and the consistent results obtained between runs makes it possible to reduce the wall thickness of the castings. The resultant saving in material, together with the increased speed of operation made possible by the use of vacuum leads to lower production costs and makes the die castings economically competitive with stampings. A further advantage is that the aluminium castings can be anodised. In the 400–ton machine described a two-part hood is arranged so that one half covers the moving platen and the other half the fixed platen of the machine. As the platens are brought together the two halves of the hood meet and seal while the die faces are still some distance apart. At this point the system is evacuated by an accumulator storage tank. The die continues to close up and is fed with metal in a completely evacuated system. In the hot chamber machine used for zinc alloy castings a valve is built over the port through which metal is fed into the charging cylinder of the injection system. This valve closes the port during the evacuation period to prevent metal from being accidentally drawn into the dies. When the required vacuum has been obtained the metal shot is made and the vacuum is then released by opening an atmosphere intake valve. A vacuum of 15–18 inch. has been found suitable for commercial use. Little benefit is obtained by the use of lower pressures. The large valves and orifices used give a fast evacuation cycle which does not slow down the operation of the machine. Elimination of entrapped air reduces the blistering of hot castings to a minimum and vacuum castings can therefore be ejected from the dies after a shorter cooling period than is otherwise necessary. This permits an increase in cycle speed which may be as high as 25% of the overall cycle time of the machine. Since vacuum castings can be heated without forming surface bubbles it may prove possible to heat-treat aluminium die castings to improve their mechanical properties. Aluminium and magnesium die castings are produced in a cold-chamber machine, in which the vacuum is also used to draw the metal directly from the furnace into the charging cylinder. In this way the charging cylinder can be filled in well under a second without any temperature loss or exposure to the atmosphere. Because of this it is possible to dispense with the silicon additions which are normally made to aluminium alloys to overcome casting difficulties due to heat losses. Freedom from silicon and oxide inclusions makes the castings easy to anodise and surface finishes comparable to those on anodised aluminium sheet or extrusions can be obtained.

201/I

Paper by A. F. Bauer *Light Metals* 20, August 1957

Large Aluminium Die Castings

United States. New developments in aluminium die casting, which in America has recently produced an output greater than the combined tonnage of aluminium sand and gravity castings, are described. Doehler 2,000–ton die casting machines with locking pressures of 1,000–1,250 tons are in production and can be used to cast 8–cylinder engine blocks weighing 55–65 lb. Mechanical locking devices such as toggle links and wedge lock systems are used on these large machines. The direct hydraulic locking system has not been used on machines heavier than 800 tons. Automatic pouring devices developed for use with the larger die-casting machines include the 'Ajaxomatic' unit whereby the metal is poured into the shot sleeve through a ceramic pipe which is submerged in an induction-heated metal bath. Pressure on the surface of the molten metal forces the metal through the pipe into the shot sleeve and an adjustable timer controls the amount of metal poured. An automatic ladling unit designed by the Lindberg Engineering Co., operates on the principle of a pressurised chamber, the size of the shot depending on the amount and duration of the pressure applied. An automatic metering device for magnesium for use with horizontal cold chamber machines has been developed by the Dow Chemical Co. A large die is difficult to produce as a forged impression block and a split-design, which is less subject to temperature stresses, is preferred. Die evacuation as an aid to pressure die casting has been hampered by the difficulty of maintaining vacuum seals, especially on intricate dies with side cores. A recent development is to enclose the casting die in a metal box which is sealed and evacuated every cycle when the die is closed. The box acts as an accumulator during the shot and extracts gas which is generated by vaporised lubricants or other sources. With this method a partial vacuum of 18 inch. mercury is obtained. A better vacuum is extremely costly to maintain. In one vacuum method under development for use with cold-chamber machines, the molten metal is sucked out of a metal pot through a vertical tube into the shot sleeve. It is believed that suitable vacuum equipment will be produced for aluminium cold-chamber machines but it is difficult and costly to maintain in the die a vacuum of less than 15 inch. Hg. Under these conditions the air in the cavity is only partially evacuated, so that porosity caused by entrapped air can only be reduced, not eliminated. Shrinkage voids in heavy sections are not reduced by the vacuum system. The higher cost of operating a vacuum process may be justified on aluminium castings which must meet extremely high surface finish or soundness requirements.

202/I

A Device for Tensile Tests of Irradiated Metals

United States. The apparatus here described was designed to facilitate tensile testing of highly radioactive specimens at temperatures up to 800°C. The unit can measure elongations up to 50% with a 1 inch sample and is fitted with an optical system for strain observations *via* a shielding window. The vacuum system was

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designed as an integral part of the apparatus to prevent oxidation of samples at high temperatures. Special requirements of the vacuum furnace were as follows: (1) Uniform temperature distribution in heating chamber. (2) Quartz windows to allow the use of optical equipment. (3) Special seals to maintain vacuum during operation. (4) Remote control for safety of operation. The test sample in the form of a circular-section rod, thickened at each end was held in pull-rods, attached to spherical seatings in the cross heads of the testing machine. The furnace was constructed in the form of a cylinder coaxial with the sample. Loading was applied to the sample *via* two sets of vacuums bellows one at each end of the furnace chamber. Heating coils were placed symmetrically around the sample and parallel to its length in the form of four small tubes so that the temperature difference in the specimen region was $\pm$ 1°C. The temperature was measured by thermocouples brought out *via* a vacuum seal activated by an air cylinder mounted on the top flange of the upper bellows. Remote control was obtained using three horizontally placed air cylinders to break the vacuum seal between the upper bellows and the furnace itself. The furnace could then be lowered to facilitate remote handling of specimens. Viewing of gauge marks on the specimen was carried out *via* quartz windows level with these marks. A novel arrangement was for the furnace to travel upwards at half the speed of the specimen pull-rod. This gives a greater range of elongation measurement than would otherwise be possible. The whole system was enclosed in a 6 inch. thick lead brick cell and viewing was done through an optically flat lead glass window. The required system pressure was 10^{-5} mm. Hg at a temperature of 800°C. The diffusion pump used was an NRC H4–P purifying-type oil diffusion pump with an ultimate of 4×10^{-7} mm. Hg. This was backed by an NRC–4S rotary pump. The diffusion pump speed was 300.l./sec. at 1μ pressure and this served to maintain the required degree of vacuum. An ion gauge and a thermocouple type gauge were used to measure the pressure and temperature respectively and were placed as close as possible to the furnace. All valves were controlled from a single panel. The pumping line to the furnace was a 36 inch. long bellows which passed through the lead brick wall and gave enough play to allow for raising and lowering the furnace. On test the maximum thermocouple differences were 1°C and temperature fluctuations were $\pm$ 1°C.

Article by
R. E. Hueschen
D. C. Kaulitz
Nucleonics
15, March 1957
88-92

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Effect of Oxide Surface Layers on Creep

203/I

United States. In the investigations described here single crystals of tin were exposed to ethyl alcohol vapour under reduced pressure. Creep data was obtained both with oxide film (2,000 Å) present and after its removal by cathodic bombardment in a discharge tube. Specimen sizes were about 0.5 mm. diameter and 20 mm. long. Data obtained with a creep stress of 275 g. per sq. mm. (390 p.s.i.) are shown in graphs. Besides vacuum and ethyl alcohol vapour the crystals were tested in various other vapours. Non-polars such as pentane had no effect. Ethylamine caused a maximum in creep rate with increasing pressure. Water vapour caused an increase proportionate to pressure. Interpretation of results leads to the conclusion that the oxide layer on crystals facilitates creep because of the creation of non-uniform stress states.

Article by
Anon.
Metal Progr.
Nov. 1956
156, 158, 160, 162

Oxygen Content of Silicon Single Crystals

204/I

United States. The oxygen content of pulled silicon crystals, determined by vacuum fusion gas analysis, is correlated with the infra red absorption at 9μ. In vacuum and in helium or hydrogen of 1 at., the oxygen concentration of a liquid zone of silicon (about 20 mm^2 in cross section) can be rapidly decreased. In an oxygen atmosphere or in contact with quartz, liquid silicon picks up oxygen quite readily. A maximum oxygen content of 2×10^{18} oxygen atoms per cm^3 was observed. Silicon single crystals pulled from a quartz crucible exhibit fluctuations in oxygen concentration both perpendicular and parallel to the pulling direction. Explanations of these effects are offered and the influence of various pulling parameters is discussed.

(*Authors*)

Article by
W. Kaiser &
P. H. Keck
J. Appl. Phys.
28, August 1957
882-887

Thermal Restoration of Oxygenated Germanium Surfaces

205/I

United States. Cleaved germanium surfaces, oxygenated at room temperature, were heated under high vacuum and the restoration of their oxygen-adsorbing capacity was determined. No restoration was observed at 425°C. Above 575°C, however, the oxygen-adsorbing capacity of the surfaces was completely recovered. Discharge of GeO from the surface was associated with this process. The restoration process followed first-order reaction kinetics with an activation energy of 56 kcal/mole. Concurrently with restoration a pronounced decrease in the surface area was observed. The results are discussed in terms of the kinetic and energy requirements of the system.

(*Authors*)

Article by
A. J. Rosenberg,
P. H. Robinson,
& H. C. Gatos
J. Appl. Phys.
29, May 1958
771-775

An Apparatus for Investigating the Low-Pressure Oxidation Kinetics of Hot Metals

206/I

United Kingdom. The apparatus was designed for the study of the oxidation of hot titanium at pressures usual in technological vacuum practice (of the order of 1μ Hg) and is believed to have some novel features, particularly the attainment of isobaric conditions with sorption rates of about 100μ litres per minute. Because of this high sorption rate a continuous flow technique was evolved so as to avoid errors due to pressure changes in the reaction vessel if it were isolated from the source of gas whilst observations were made. The requirements aimed at were: continuous observation of oxidation rate; constant oxygen pressure; avoidance of accumulation of gaseous impurities in the reaction vessel; no restriction on the higher limit of metal temperature by the materials of the apparatus; physical properties of the metal to be observable during oxidation; independent variation of temperature of the gas and the metal. All these requirements were in the main achieved by the present method. The reaction vessel is of Pyrex, transparent silica, or gold-plated copper (the latter having a

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protected Pyrex window) and contains the metal under test in the form of a 30–40 cm. long hairpin filament, and is provided with a Pirani gauge and an optical pyrometer. A liquid nitrogen trap and ionisation gauge are mounted close to the reaction vessel. The rest of the apparatus is constructed of Pyrex glass. A manostat maintains a constant pressure of oxygen in a graduated gas-holder from where it is fed to a T-junction where another Pirani gauge is mounted. One arm of the junction is connected to the reaction vessel, the third arm is connected, *via* a variable throttle, to an oil-diffusion pump which removes the surplus oxygen and impurities. The system was pumped for a week to remove impurities, a constant overnight accumulation of about $1/25\mu$ Hg (achieved after four or five days) was taken as an empirical indication of the cleanness of the system. The filament was heated electrically to the required temperature and pressures and flow rate measured. The heating current was switched off and the flow rate adjusted to give the same pressure reading in the pump arm. From these observations the oxidation rate was calculated. The probability of reaction of oxygen with the hot metal was found to be of the order of unity for titanium at a temperature of 1,000°C and 1μ Hg pressure. The apparatus is also suitable for continuously introducing a metered gas flow of the order of 100μ litres per minute into a low pressure system.

Article by L. G. Carpenter & W. N. Mair *J. Sci. Instrum.* **34**, March 1957 110-114

207/I

Reaction Between Oxygen and Hot Gold

See Abstract No.: 21/I

208/I

Oxidation of Barium: A Confirmation of Mott's Theory of Oxidation

See Abstract No.: 53/IV

209/I

Vacuum Fusion Determination of Oxygen and Nitrogen in Lanthanum

See Abstract No.: 160/III

210/I

Determination of Oxygen in Niobium

United States. Since niobium is being used to an increasing extent in nuclear and other applications, it has become essential to determine accurately the gas content of the metal because this affects its physical properties. This paper reports an investigation into methods of determining the oxygen content of niobium. After preliminary work had eliminated some methods which gave low and erratic results it was found that a diffusion-extraction method using conventional vacuum-fusion equipment gave satisfactory results. The mean deviation of analyses of 0.5 gram. samples containing 0.019 weight % oxygen was $\pm$ 0.001 weight %. The sensitivity of the analysis could be increased by increasing the sample size.

Article by W. R. Hansen & M. W. Mallett *Analyt. Chem.* **29**, December 1957 1868-1869

211/I

Columbium and its Uses

United States. Columbium, or niobium, has properties which make it of value in nuclear energy and high temperature equipment. Its potential usefulness has been increased by the discovery of large reserves of ore and by the development of a new method of separating columbium from the tantalum with which it is usually associated in nature. The metal has a medium density of 0.31 lb./cu. inch, about half that of tantalum. It has good strength at high temperature and, unlike molybdenum, retains its ductility after heating to high temperature. The oxidation resistance is poor compared with that of conventional heat-resisting alloys, but is better than that of molybdenum and the metal does not suffer catastrophic failure by oxidation at high temperature. Thus it would not fail immediately on the failure of its protective coating. Alloying with zirconium shows promise as a means of improving oxidation resistance. The metal is of particular use for fast reactors because of its high temperature strength and nuclear characteristics. It has a low thermal cross section and is resistant to liquid sodium and bismuth which may be used as coolants. It has been chosen as a canning material in the Dounreay reactor. Columbium can also be used as an alloying element to stabilise the protective oxide film on uranium. An alloy containing about 7% columbium is satisfactory, even in oxygen-free water, at temperatures as high as 660°F. The properties of columbium are greatly influenced by gas absorption during processing. Its strength and hardness increase and its ductility decreases as the oxygen content increases. For good fabrication properties the oxygen content must be held below 0.03%. The oxide formed on columbium is adherent but dissolves in the metal on heating in a vacuum between red heat and 2,200°F. However it has been found possible to hot work columbium at 1,800°F and obtain ductile material. Sintered bars of columbium can be cold-rolled to sheet and the metal can be drawn, stamped or formed into complicated shapes by cold working. Machining can be carried out with ordinary tools. With the exception of its lower density and easier fabricating characteristics, the properties of columbium are similar to those of tantalum. Columbium has slightly higher thermal expansion and electrical resistivity and lower thermal conductivity. Its corrosion resistance is good, but lower than that of tantalum. Columbium is at present produced commercially by extracting columbium oxide from the ore and reducing this oxide with columbium carbide in a vacuum. In the future it may be possible to reduce columbium pentachloride by magnesium or sodium. The carbide reduction yields metallic columbium in powder form which is subsequently consolidated by sintering or melting. Sintering is generally employed because it combines consolidation with purification. Consolidation may be effected by induction or direct resistance heating in a vacuum. The best results are obtained if the powder is first heated in vacuum at 2,000°F to remove hydrogen and then blended with coarse powder produced by

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crushing and degassing columbium scrap that has been embrittled by hydriding. Oxygen can be removed by volatilisation as a lower columbium oxide at 3,800 to 4,200°F or, preferably, by the interaction of oxygen and carbon at 3,100 to 3,450°F to form carbon monoxide. In practice, for the latter method, the furnace is evacuated to a pressure below 10^{-4} mm. Hg. The temperature is increased to the range 3,100–3,450°F in 40 min., held for about three hours until most of the carbon and silicon monoxides are removed, and then raised gradually over six hours to a temperature of 4,200°F. This temperature is maintained for six hours to remove impurities and obtain sufficient degassification to yield a ductile product. In another, alternative method, the degassing period at 3,100–3,450°F is cut short and the partially sintered, brittle bar is crushed, recompacted and resintered.

Article by G. L. Miller *Materials & Methods* May, 1957 131-135

Improvements i.o.r.t. the Production of Niobium 212/I

United Kingdom. The production of niobium powder in quantities of about two kilogrammes by reduction of the fluoride, potassium niobium, is described. Reduction is accomplished by heating the fluoride together with a stoichiometric excess of about 10% of sodium metal in an inert atmosphere until the resulting reaction is completed. The products are then heated to a temperature above the boiling point of sodium to distil off excess of that metal and any potassium formed. After cooling, the reaction products are crushed and leached with considerably more water than is necessary to remove all the sodium fluoride formed in the reaction. At least two leachings are advisable, the first being completed in a short period, for example three minutes, in order to limit the loss of niobium as niobate. The apparatus used comprises an electric furnace, a stainless steel reactor vessel 9 inch. in diameter and 3 ft. 9 inch. long, and a mild steel crucible 8 inch. in diameter and 2 ft. 6 inch. high. The top of the reactor vessel is connected to a water-cooled condenser designed to prevent condensed metal returning to the reactor. The top of the condenser is connected to a conduit fitted with a valve which can be opened to the atmosphere, and terminating in one of a pair of valves provided with gas-tight coupling means. The second valve of the pair is carried by a conduit leading to an expansion chamber. This chamber is fitted with pressure gauges and can be connected to an evacuating system and a supply of inert gas. In the example quoted, 5.72 kg. of potassium niobium fluoride and 2.7 kg. of pure sodium metal pellets are rammed into the crucible in alternate layers of sodium and fluoride, starting with a layer of 5% of the sodium pellets. The charged crucible is lowered into the reactor vessel, the condenser and conduits are connected, and the system is evacuated and filled with argon to atmospheric pressure. Evacuation and filling with argon are repeated twice. With the reactor vessel in position, the furnace is heated to 400°C and held at this temperature for half a hour to ensure that the whole charge is heated. The temperature is then raised until reaction commences at about 420°C or above. The pressure rise which accompanies the reaction is indicated on a gauge. When the reaction has ceased, the temperature is raised to about 1,100°C for at least one hour. During this period the condenser is cooled to condense sodium vapour and any potassium vapour. Heating is then stopped and when the reactor has cooled to 600–700°C, it is sealed and disconnected from the second conduit, removed from the furnace and allowed to cool to room temperature in air. It is then opened to the atmosphere, the condenser is disconnected and the crucible is removed. The reaction products, which form a cake, are mechanically crushed to approximately 20 mesh size. The granular product is leached five times and is then transferred to a filter and washed with de-mineralised water. The resulting powder is dried. Yields of 90% of the niobium can be obtained. The particles of niobium produced have sizes mainly in the range 5 to 66μ. They have been compacted and then sintered at 2,300°C to give a bar of hardness 130 to 150 V.P.N.

U.K. Atomic Energy Authority *Brit. Pat.* 791,121

Zone Melting of Uranium 213/I

See Abstract No.: 164/III

Uranium and Its Alloys 214/I

United Kingdom. The properties of uranium which affect its use as a nuclear fuel in thermal reactors are discussed with reference to the influence of alloying, fabricating processes, thermal cycling and neutron bombardment. Uranium can exist in three allotropic forms: An isotropic gamma phase between 769°C and the melting point; an anisotropic alpha phase, stable up to 662°C; and a beta phase, stable from 662° to 769°C, which is also anisotropic to a certain extent. Many of the difficulties in the use of uranium are due to the anisotropic nature of alpha uranium, and it is desirable to use fine-grained, randomly-orientated material to counteract the directional properties of the crystals. Certain physical properties, such as density and vapour pressure, are independent of the exact purity of the metal and of the treatment it has received. Others, including elastic moduli, thermal expansion and mechanical strength are influenced by the directional properties and hence by processing methods. The mechanical properties are highly dependent upon purity and upon the fabrication and heat-treatment histories, and the values obtained vary widely between specimens. The creep strength of uranium depends on its composition and grain size. Creep tests must be carried out under good vacuum or inert atmosphere conditions and temperature variations during a test must be kept to a minimum. The low stresses and slow strain rates experienced under reactor conditions are difficult to reproduce experimentally. In Britain nuclear fuel elements are generally made of machined and heat-treated bars of cast uranium. The metal is commonly melted in vacuum h.f. furnaces with graphite crucibles and moulds, but alloys of high melting point may require the use of uranium oxide or beryllia crucibles to avoid excessive carbon pick-up. Alternatively the alloys may be arc-melted. All the common working practices are applicable to uranium, but rolling and extrusion are the only processes used to a large extent. Uranium parts can be produced by powder metallurgy using powder prepared by decomposition of the hydrides. By pressing the

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cold powder at 70 ton/inch.^{2} and sintering in vacuum at 1,125°C a compact with 94% of the theoretical density is obtained. Hot pressing in vacuum at about 20 ton/inch.^{2} and a temperature of 600°C gives material of slightly higher density with a finer, randomly oriented grain structure. An extrusion process has also been developed. Difficulties in machining uranium are caused by the rapid work hardening and oxidation of the metal. Welding and brazing are hampered by the reactivity of the metal at high temperatures, but some success has been achieved with pressure welding in an argon atmosphere using high frequency heating. Operating conditions which entail repeated heating and cooling through a temperature range in which a phase transformation occurs, or thermal cycling in the alpha range, give rise to a change in shape which causes wrinkling of the metal surface. This effect may be counteracted by using a strong container or by alloying to avoid or reduce the influence of the beta phase. Dimensional effects which give rise to surface wrinkling are also caused by neutron bombardment in a reactor. The fine grain structure necessary to reduce this effect may be achieved by traverse quenching which consists of passing the bar through an h.f. heating coil and then through a water spray. Another result of the anisotropic growth of uranium crystals under neutron bombardment is an increase in the rate of creep. Like the irradiation distortion which causes them, irradiation creep and wrinkling are expected to reach a maximum at about 200°C and then decrease with increase of temperature to zero about 400°C. At higher temperatures gaseous fission products give rise to another form of swelling. Many of the properties of uranium, including hardness, thermal conductivity and corrosion resistance are also affected by irradiation. Although alloying additions can improve certain properties their use may entail disadvantages such as increased difficulty in processing the element after irradiation.

Article by R. W. Nichols *Nuclear Engng.* **2**, Sept. 1957 355-364

215/I

Preparation of Uranium Metal

United States. A process is described for producing solid uranium metal deposits of high purity upon a heated refractory body, such as a tungsten filament, by decomposition of uranium tetraiodide vapour. The process is carried out in an oven-heated reaction vessel, for example a Pyrex flask, which can be evacuated to a very low pressure. The filaments are supported in the vessel in the usual way. The tetraiodide vapour is formed by reaction between uranium triiode and iodine vapour. A charge of uranium triiode is introduced into the reaction vessel *via* a feed tube which can subsequently be sealed off. The charge falls into an annular well which forms the lower part of the reaction vessel and this well is surrounded by an independent heating and/or cooling device so that its temperature can be varied from that of the main part of the vessel. Iodine vapour is introduced through a vertical tube which is hermetically sealed into the base of the reaction vessel. The tube passes through the annular well to a level above that normally reached by the UI_3 charge, and its upper end is attached to an inverted bell cap which directs the flow of iodine vapour into the reaction vessel. The part of the tube just below the well can be heated by an auxiliary oven to prevent deposits forming on its walls. The lower end of the tube contains a frangible capsule of iodine which can be broken when required by an iron weight controlled by a magnet. The iodine reservoir is surrounded by a heating and/or cooling device so that the temperature of the iodine, and thus the rate of vapour formation, can be controlled. The amount of iodine used is sufficient to convert the trivalent uranium content of the UI_3 charge to tetravalent uranium according to the reaction: $UI_3 + \frac{1}{2}I_2 \rightarrow UI_4$. The apparatus is given a preliminary bake-out, after which the charge of UI_3 is admitted in an atmosphere of inert gas. A second bake-out is carried out during which the tungsten filaments are degassed by flashing. The reaction vessel is then maintained under vacuum and heated and the iodine vapour is admitted. Under suitable operating conditions tetraiodide vapour is formed which decomposes upon contacting the extremely hot filaments in accordance with the reaction: $UI_4 \rightarrow U + 2I_2$. The success of the method depends to a large extent upon controlling, within certain critical limits, the partial pressure of the iodine present in the reaction zone, and the temperature of the filament. Iodine partial pressure is controlled by the temperature of the iodine source and by the vacuum applied to the system, and should be within the range 0.003–0.10 mm. Hg. The filament must be kept above about 800°C, but below the melting point of uranium. The triiodide and free iodine react at a practicable rate over the temperature range 400–800°C. The temperatures of the main and auxiliary ovens should be kept about 30° higher than that of the annular well to prevent any deposits forming on the walls of the apparatus. Operating conditions which give good results are as follows: filament temperature 1,032–1,107°C; iodine partial pressure 7×10^{-3} mm. Hg (corresponding to about −12°C in the iodine reservoir); temperature of the reaction vessel and vertical tube 520–560°C; temperature of the UI_3 in the annular well 500–530°C. Pumping pressure on the reaction vessel, 10^{-6} to 10^{-7} mm. Hg. The body on which the uranium metal is deposited may be a refractory other than tungsten, for example tantalum, molybdenum or platinum. It may also be constructed of uranium itself in which case a solid bar of substantially pure uranium can be built up. If required, the refractory body may be coated with a refractory oxide and it may be in some form other than a filament, for example a plate, disc or tube.

C. H. Prescott, J. A. Holmes & U.S. Atomic Energy Comm. *U.S. Pat.* 2,785,972

216/I

Apparatus for Vacuum Casting of Uranium

United States. In the process described uranium metal containing impurities internally and/or externally in the form of a surface skin is melted under conditions which enable the molten metal to be withdrawn away from the solid impurities. Most of the volatile impurities are also removed in the melting chamber at temperatures of 1,200–1,500°C. In order to minimise the formation of uranium carbide the temperature should preferably be kept below 1,500°C and the heating period should be as short as is compatible with the removal of the volatile impurities. Before being cast into carbon moulds the metal is allowed to cool to slightly above its melting point, which has been found to be in the neighbourhood of 1,100°C and not about 1,800°C as was once thought. Most of the uranium carbide formed in the melting chamber remains there as part of the retained solid impurities. Pouring at temperatures as low as 1,100–1,300°C reduces the intensity and duration of reaction between the molten uranium and the carbon mould so that contamination of the metal and pitting

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of the mould are restricted. The melting crucible is cylindrical and is covered with a lid designed to permit the escape of volatile impurities whilst preventing them from impinging on the outer parts of the apparatus. The bottom of the crucible slopes to an aperture of ¼–2 inch. dia. sealed by a plug. When the plug is raised molten metal can pass down the aperture into a mould situated immediately below the crucible and containing one or more casting chambers of the required shape. Elements of the apparatus which come into direct contact with the uranium are preferably made of graphite but may be of any suitable refractory. One advantage of a machined graphite crucible is that impurities adhere to its surface as the molten uranium flows through the aperture. The mould rests upon a solid block provided with channels for evacuating the system or introducing inert gas. This block in turn is supported on a base which is preferably a water-cooled brass casting. The plug sealing the bottom of the melting chamber is dislodged by means of a trip rod which passes through vertically aligned channels in the base, supporting block and casting chamber. An airtight shield envelops the apparatus. Heating is effected by an induction coil which surrounds the shield and which can be raised or lowered to provide heat at any required position. Temperature measurements are made by thermocouples inserted at suitable points. Heat losses from the melting chamber and the upper portion of the mould can be reduced by insulation, preferably in the form of curved sheets fabricated from thermal-setting sillimanite and sawdust. In order to prevent unseating of the plug during the vacuum process, a small amount of finely divided uranium compound is placed over the plug followed by resilient turnings of uranium metal. The load of ingots upon the turnings then keeps the plug in position. When the apparatus has been charged and evacuated the induction coil is arranged so that the bottom of the coil is about one inch below the bottom of the crucible and the temperature of the crucible is raised to 1,000°C for 8–12 minutes and then to about 1,360°C for 10 minutes. The uranium is then cooled to about 1,200°C and the plug is unseated so that the metal pours into the casting chamber. The induction coil is lowered about three inches and operated for about 10 minutes. By gradually raising the coil during this period the cooling of the cast metal can be regulated so that solidification begins at the bottom of the casting chamber and proceeds upwards. In this way pipes or hollows in the upper surface of the casting may be prevented. Finally the apparatus is allowed to cool, the vacuum is released and the apparatus disassembled for the removal of the castings.

H. A. Wilhelm, C. F. Gray & U.S. Atomic Energy Comm. *U.S. Pat.* 2,782,475

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A High-Vacuum Casting Furnace for Plutonium

217/I

United States. A high-vacuum casting furnace with an internal diameter of 15 inch. is described. It will accommodate casting crucibles up to 7 inch. o.d. and with a larger diameter heating element, a crucible of 9 inch., o.d. The furnace is heated by two parallel 10-ohm 0.050 inch. tungsten wire coils wound on the outside of a 9-inch. grooved alundum sleeve. The unit will heat up to 1,000°C in two hours with both legs drawing 25A at 80–90V. A complete set of engineering drawings of the furnace is given.

Report by J. W. Anderson *Univ. Calif. Los Alamos Lab. Rep.* LA-1508

Coating a Carbonaceous Surface with Rhenium

218/I

United States. The value of graphite as a structural material for high temperature use is limited by its brittleness, high porosity and poor surface hardness. It also has comparatively low corrosion resistence to many liquid metals. These disadvantages can be overcome by bonding a metallic coating to the surface of the graphite structure. Such a coating should have a very high melting point, should be easy to apply and should remain strongly bonded to the graphite surface at high operating temperatures. Rhenium, which has a melting point of over 3,000°C, is a suitable metal. A rhenium inner coating on graphite tubes and other components of fluid circuits provides a hard metallic finish which resists corrosion and prevents leakage. Rhenium-coated graphite wire is useful for emitter elements, particularly in x-ray tubes. These elements are structurally strong at high temperatures and are preferred to tungsten elements because of the lower volatility of rhenium, and hence longer life of the elements. Coating of the graphite object is effected by electroplating followed by heating in vacuum to a temperature of at least 1,100°C. A rhenium-to-graphite bond of high strength up to temperatures of 1,000°C is obtained. Prior to plating the graphite is cleaned and oven-heated to remove moisture and gases from the pores. One suitable electrolyte is a sulphuric acid solution containing potassium perrhenate with a solution pH of 0.9. Plating is carried out at a solution temperature of 25–50°C using a voltage between the electrodes of 5–10V and a current density of 1–15 A/c.m. of surface area of the graphite object. The plating is made thicker when surface actants of the glycol type, such as Carbitol, are added to the bath. Another suitable electrolyte is a 10% potassium perrhenate solution with sufficient citric acid to form a solution with pHl. With this bath plating is accelerated and depth of plating increased by actants such as triethanolamine or sulphurous acid. After the plating has been completed the coated object is washed in distilled water and dipped in anhydrous methyl alcohol to remove excess moisture. It is then heated in an air oven to 110°C to evaporate the methyl alcohol and so reduce the load on the vacuum pump in the final baking stage. The coated object is not left in the air oven for more than a few hours or the rhenium oxidises and the strength of the coating is reduced. After this heating step, the coated object is transferred to a high-temperature furnace where it is heated to a high temperature in a vacuum. The best grain structure is obtained by heating to 2,200°C although satisfactory results have been obtained at temperatures as low as 1,100°C. The graphite may be heated by induction or other types of furnace may be used. This final heating process improves the grain structure of the rhenium plating forming a smooth non-porous coating on the graphite base. It increases the strength of the rhenium-to-graphite bond and removes any residual sulphuric acid. For additional thickness of the rhenium coating the plating-heat treatment cycle may be repeated as often as desired.

H. Pearlman L. Silverman C. A. Smith & U.S. Atomic Energy Comm. *U.S. Pat.* 2,778,786

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219/I

Molybdenum Alloys: When to Use Them

United States. The properties and uses of four arc-cast molybdenum alloys, which are now commercially available, are compared with those of the unalloyed metal. The alloys contain respectively: 0.3% columbium; 0.5% titanium; 1.0% vanadium; 2.0% tungsten. Of these, the titanium alloy appears to have the best all-round combination of properties. It has greater structural strength at temperatures above 1,600°F than any other commercially available material. Alloying elements have little effect on the physical properties of molybdenum which is characterised by its high melting point, high thermal and electrical conductivity, low expansivity and high modulus of elasticity. Alloying, however, improves the high-temperature strength and hardness and raises the re-crystallisation temperature. A high-recrystallisation temperature is desirable since the alloys are not heat-treatable. They are strengthened by mechanical work which loses its effect when the metal re-crystallises. The alloys are preferable for applications such as hot working tools and dies, accessories for glass-melting furnaces and gas turbine buckets and guide vanes. Unalloyed metal is suitable for electronic components, electric furnace parts and heat exchangers. It has been used for the heating gauge and radiation shields of Balzers high-vacuum sintering furnace. Forming and machining practices are similar for both the metal and its alloys. Welding properties of arc-cast metal are better than those of molybdenum prepared by commercial powder metallurgy. At present the most satisfactory welds in heavy sheets and parts are made by the inert-gas-shielded arc process using tungsten electrodes with or without filler wire additions. Unprotected molybdenum cannot be used continuously in air or oxidising atmospheres at temperatures above 1,000°F because of its rapid oxidation. Methods of surface protection have been devised on a laboratory scale and some of these may prove suitable for commercial use, especially where a useful life of 500–1,000 hours is acceptable. Three promising methods are cladding, electroplating and spray coating. Techniques developed for the unalloyed metal appear suitable for its alloys. One difference which has been observed, however, is that, in the diffusion treatment used to bond a metallised coating, an argon atmosphere seems more suitable for alloys than the hydrogen atmosphere commonly used for unalloyed molybdenum.

Article by R. R. Freeman & J. Z. Briggs *Mater Meth.* November 1956 114-117

220/I

Recent Studies on Ductile Molybdenum

United States. A study has been made of the effect of impurities on the room-temperature ductility, high-temperature embrittlement and welding properties of molybdenum. Powder metallurgy bar stock, which has a lower carbon content than arc-cast molybdenum, was used as the starting electrode material and was refined by repeated melting under high vacuum. Some high-purity ingots were obtained with an oxygen content as low as 0.0001%. Other impurities were carbon 0.002%, nitrogen 0.0002% and hydrogen 0.00005%. Specimens were contaminated for testing by introducing oxygen and nitrogen into the furnace at low pressure during final melting. Carbon was added as a master alloy in the original electrode. Molybdenum wire and rod were purified by solid-state purification. With this method, by heating above 1,900°C, but below the melting point, in a vacuum of 0.1μ Hg for 6 hours it was possible to reduce the oxygen to 0.0003% and the nitrogen to 0.0002% in a sample previously decarbonised by heating in flowing oxygen at 1,650°C. An unnotched bend test showed that minute amounts of oxygen (0.0002%) cause severe embrittlement in cast molybdenum. With nitrogen, brittleness occurs at about 0.003%, and, with carbon, at about 0.014%. Brittleness is attributed to precipitates of oxides, carbides or nitrides at the grain boundaries. When sufficient to produce networks the precipitates start and propagate cracks between ductile crystals. Heating in vacuum puts the precipitates into solution and after quenching the ductility is improved. Solid-state purified rod with an oxygen content of 0.0003% oxygen was found to be ductile even when fully re-crystallised. In one experiment a wire, 0.04 inch. diameter, consisting of a single crystal, was heated alongside a polycrystalline wire in a low pressure of oxygen sufficient to embrittle the latter. The single crystal remained ductile, despite the presence of oxygen in solid solution, because there were no grain boundaries in which a second phase could collect. Impurity elements in solid solution, however, may increase the tendency to transgranular brittleness. The loss of ductility when a cold-worked piece is heated for re-crystallisation is attributed to contamination from the furnace atmosphere and not to any effect of the re-crystallisation process. The so-called 're-crystallisation embrittlement' is thus a misnomer. Since it is difficult to prevent contamination of high-purity molybdenum during fabrication, attempts have been made to find elements which can be added to the metal to convert the precipitates into innocuous forms. Additions of titanium have been the most effective in improving room temperature ductility and lowering the transition temperature. Molybdenum-titanium alloys also appear to give the best welding properties. The oxygen content of commercial powder-metallurgy molybdenum is usually sufficient to embrittle welds even when arc-welded in purified inert atmospheres. Welds in commercial molybdenum of low oxygen content, carbon de-oxidised and arc-cast are embrittled by carbide precipitation.

Article by J. J. Harwood *Metal Prog.* Dec. 1956 97-101

221/I

Process of Smelting Germanium

See Abstract No.: 168/III

222/I

Thermionic Constants and Sorption Properties of Hafnium

See Abstract No.: 70/IV

223/I

Method of Producing Metals by Decomposition of Halides

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Improvements i.o.r.t. a Process and Apparatus for the Production of Thin Deposits upon a Support by Decomposition of a Gaseous Material — 224/I

See Abstract No.: 192/III

Problem of Gases in Metals — 225/I

See Abstract No.: 47/I

Process for the Degassification of Molten Steels — 226/I

See Abstract No.: 152/III

Mass Spectrometer Measurements of the Diffusion Coefficient of Hydrogen in Steel in the Temperature Range of 25°–90°C — 227/I

United States. The diffusion coefficients for hydrogen in mild steel have been measured in the temperature range of 25° to 90°C. A mass spectrometer was used to study the movement of hydrogen through thin steel plates. Two methods of measuring diffusion have been used; the rate of approach to equilibrium when hydrogen is supplied to the plate, and the rate of outgassing when the source of hydrogen is removed. Two decidedly different sets of diffusion coefficients were obtained by the two methods. The first method gave diffusion coefficients which agree very well with values obtained by others at higher temperatures while the outgassing method which was more reproducible, gave values considerably lower. These results are compared with those of other investigators.

(*Authors*)

Article by R. C. Frank, D. E. Swets & D. L. Fry *J. Appl. Phys.* **29**, June 1958 892-898

Ratio of the Diffusion Coefficients for the Diffusion of Hydrogen and Deuterium in Steel — 228/I

United States. The ratio of the diffusion coefficients for hydrogen and deuterium in steel have been measured in the temperature range of 26°C to 86°C. A mixture of the two gases was introduced into the steel by abrasion in a mixture of normal and heavy water. The diffusion coefficients were measured simultaneously for the two using the mass spectrometer to observe the rate of evolution of the gases from the steel. The ratio of the diffusion coefficients was found to be constant within the limits of experimental error over the entire temperature range and had a value of 1.37 ± 0.02. The ratio of the permeation rates was found to vary with the temperature. The results are compared with those of other investigators.

(*Authors*)

Article by R. C. Frank, R. W. Lee & R. L. Williams *J. Appl. Phys.* **29**, June 1958 898-900

Transformer Steel — 229/I

United Kingdom. Experiments by Lihl have confirmed that when steel is given a final annealing in a high vacuum the demagnetisation losses are smaller than when a protective atmosphere such as a nitrogen-hydrogen mixture or pure hydrogen is used. The experiments consisted of magnetic, metallographic and x-ray diffraction studies on hot- and cold-rolled transformer steel sheet. In the case of the hot-rolled sheet, the initial purity of the metal had less influence, and the effects of over-annealing on the non-metallic inclusions were less harmful when vacuum annealing was used. In cold-rolled sheet, grain growth and the formation of a re-crystallisation texture favourable to the magnetic properties were helped by annealing in a high vacuum. Vacuum-annealed hot-rolled sheet showed demagnetisation losses of about 0.84, 1.47 and 2.15 W/kg for inductions of 10 kg, 13 kg and 15 kg. These values are about 10% lower than the best results obtained after annealing in protective gas. In vacuum-annealed cold-rolled sheet the losses, for the same inductions, were 0.75, 1.35 and 1.9W/kg, which are considerably lower than values obtained for similar cold-rolled sheet annealed in hydrogen. Economic factors favour the use of high-vacuum annealing since it would eliminate the expense and explosion risk of pure hydrogen.

Article by by Anon. *Engineer* **204**, 30.8.57 301

Recent Developments in Vacuum Heat Treating Furnaces — 230/I

See Abstract No.: 118/III

Beryllium as an Industrial Metal — 231/I

United Kingdom. Until becoming important in the atomic energy field, beryllium was regarded as essentially an alloying metal. It was limited by cost of extraction, high melting point, high vapour-pressure, toxicity and brittleness. When found to be valuable as a moderator and reflector of neutrons, beryllium was subjected to intensive study. Vacuum melting and extrusion of beryllium was developed in the U.S. before powder metallurgy fabrication. Corrosion-resistance also makes pure beryllium important in various atomic energy applications. Other uses for the metal are in making windows which are transparent to x-rays, as thin sheet in scintillation counters and Geiger counters, as cyclotron targets and for electrodes in neon signs. Hitherto however, only one per cent of beryl ore has gone to produce beryllium metal. The various processes of extraction of beryllium ore aimed at the concentration of beryl from the ore, separation of impurities, and preparation of a form suitable for reduction. This is achieved by the Degussa electrolytic process, or the

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Kjellgren magnesium reduction method. Fabrication is also costly. Ingots are produced by vacuum melting. Vacuum casting with beryllia crucibles and graphite moulds is now well established. The difficulty of fabrication lies in the brittleness of beryllium. Sloman of the N.P.L. tried distilling the metal *in vacuo* to eliminate beryllium oxide, but no beryllium free from brittleness has yet been produced. It was discovered however that beryllium can be cold-rolled in a steel-jacket at between 370° and 1,095°C. 'Sheath-rolling' at 1,000°–1,100°C is now used for powder-metallurgical fabrication. Extrusion in a steel-jacket is also effective.

Article by M. Schofield *Light Metals* 19 Oct. 1956 312-313

232/I

Brazing Beryllium Tubing to High-Temperature Alloy Collars

See Abstract No.: 106/I

233/I

'Hortonclad' Composite Metal

Note by Anon. *Engineer* 205, 24.1.1958 152

United States. An account of the advantages of composite metal made by a high-vacuum brazing process method without the use of fluxes.

234/I

The Welding of Titanium

United Kingdom. Titanium may be welded in an inert atmosphere, under pressure, or by spot or flash welding using modified techniques. The Argonarc and Argonaut inert gas arc welding processes are described in detail. The surfaces to be welded are cleaned and freed from oxide by normal mechanical and chemical means. The argon shroud of the torch prevents contamination on the top side whilst closely fitting backing plates or an argon 'blanket' protects the underside. For the welding of component parts a special cabinet has been devised which can be evacuated to 0.5 mm. Hg by a rotary vacuum pump in five minutes prior to filling with argon. The operator wears rubber gloves which are sealed into the cabinet and views the work through a Perspex screen in the cabinet top. All welding is done in an argon atmosphere, the source being an ordinary gas cylinder. Plates up to $\frac{1}{8}$ inch thick may be butt welded but thicker sections require a 60° bevelled Vee.

Article by Anon. *Machinery* 89, 19.10.1956 915-916

235/I

Titanium and Aircraft Quality Steels

United Kingdom. The titanium melting plant of William Jessop and Sons Ltd., which came into production in 1956, produces titanium ingots up to 20 inch. in dia. and approximately one ton in weight. The original capacity of 5–7 tons a week is scheduled to be more than doubled in 1958. Five alloys under the brand 'Hylite' are in production and others are being developed. Hylite 10 is a low-strength commercially pure titanium and Hylite 50, the fifth of the series, is a complex alloy developed to give high mechanical strength and particularly good creep strength up to temperatures of 400°C. The starting material used by Jessop is granulated titanium produced by the I.C.I. sodium reduction process. Alloy additions are made to the granulated metal which is thoroughly mixed and then compacted into briquettes. These briquettes are welded together to form a 16 ft. long electrode using Argonaut welding equipment with a titanium wire electrode. The furnace crucible is sealed at the bottom with a titanium slug from the previous melting and the electrode is fed through a port and seals the top end. The arc is struck between the electrode and the base of the crucible and is maintained automatically. In order to ensure homogeneous, sound ingots with a low hydrogen content, the ingot obtained from this first melting is used as the electrode in a second melting process. Normally melting is carried out under vacuum but for certain alloys an argon atmosphere is provided. The furnaces used were designed in collaboration with an American titanium producer and the vacuum installation was supplied by Edwards High Vacuum, Ltd. Vacuum melting is also used by Jessop in the production of heat-resisting and ball-race steel alloys and ultra high-tensile steels to reduce harmful non-metallic inclusions and gases, and provide increased toughness and fatigue resistance. Using the titanium melting furnaces, steel electrodes have been arc-melted and cast under vacuum to produce ingots 20 inch. in dia. weighing about a ton. Modifications are now being made to permit the production of a heavier steel ingot. Jessop's vacuum melting equipment also includes a high frequency induction furnace in which the whole of the melting process, including alloy additions and casting, can be carried out under vacuum. This plant has been used extensively for alloy development work and is now in use for the production of gas turbine blading alloys. A similar furnace of 600 lb. capacity is to be installed. Jessop's vacuum-melted steel products are marketed under the trade name Vacumelt'.

Article by Anon. *The Overseas Engineer* October 1957

236/I

Titanium Melting Plant

Note by Anon. *Engineer* 205, 6.6.1958 865

United States. A commercial vacuum melting furnace is described.

237/I

Vacuum Furnace for Treating Titanium

United States. A furnace made by the Lindbergh Engineering Co. for the production brazing, heat-treating, annealing and stress relieving of titanium is described. The furnace may also be used to process zirconium 'cans' for nuclear reactors, and for treating other metals which require complete protection against oxidation

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at high temperatures. It is operated by North American Aviation Inc. Flat sheets up to 4 ft. by 12 ft. can be treated in the furnace which is more than 40 ft. long and has an inside diameter of 78 inch. The furnace has two internal zones. One can be heated up to 2,150°F and the other can be water-cooled to lower the temperature of a heat-treated load so rapidly that it can be removed from the furnace 30 min. after the end of a processing cycle. Alternate heating and cooling in the adjacent zones is made possible by a series of 14 vertically-mounted radiation baffles which separate the two areas and permit heat from the electrical elements to be concentrated on the required position. The furnace incorporates an Inconel inner shell separated from the mild steel outer shell by a 14-inch. thick layer of fire and insulating brick. The vacuum system can evacuate the 1,400 cu. ft. interior in 23 min. and maintain pressures as low as 0.3μ. It includes three roughing pumps, two 16-inch. diffusion pumps and one small holding pump. The battery of three transformers for the three-zone control of Lindbergh rod-type heating elements requires 820 kW. A caterpillar-type Inconel conveyor cart can handle a load of 1,000 lb. and can be moved between zones within the furnace by means of an electric motor, vacuum seal bearings and external gears. When an inert atmosphere is required gas can pass into the furnace from an external container *via* a standard high-pressure valve. A sliding furnace door which can be sealed by means of a groove and ¾-inch O-ring is operated by a chain and Acme-thread mechanism. Titanium parts are annealed by holding at 1,175°F and minimum pressure for one hour. In the brazing of honeycomb structures parts are heated to 1,350–1,725°F in an inert atmosphere and the temperature is then quickly reduced below the critical point in order to avoid microstructural changes. A temperature drop of 12°/min. can be obtained for this purpose. Ageing of solution heat-treated parts can be carried out at temperatures of 950° to 1,075°F for various periods of time and with differing atmospheres or pressures. The furnace is also used to eliminate the instability of welded titanium parts by heating them above their ageing temperatures in inert atmospheres. Adequate protection is provided for certain forms of stainless steel where annealing temperatures of 1,900°F and bright heat treating temperatures of 1,400°F are indicated. Both stainless steel and titanium alloys have been satisfactorily stress-relieved at temperatures ranging from 700° to 1,200°F.

Article by Anon. *Metal Treatment* Jan. 1958 25-26

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Arc and Vacuum Melting of Titanium: Part I — 238/I

See Abstract No.: 147/III

Arc and Vacuum Melting of Titanium II — 239/I

See Abstract No.: 148/III

Production of Titanium Shaped Castings — 240/I

See Abstract No.: 149/III

Powder Metallurgy Provides a Short Cut to Titanium Parts — 241/I

United States. The article states that although practically all the initial work on titanium was done by powder metallurgy, virtually all products are now made by arc melting followed by mill fabrication. Nevertheless, powder metallurgy may still prove important for the production of small components, porous bodies or in the development of titanium dispersion products with improved strength at high temperatures. Four methods of producing titanium powder are described. These are:—(*1*) Chemical reduction, such as the magnesium reduction of titanium tetrachloride. (*2*) Mechanical comminution by shearing, cutting or attrition mechanisms. This appears to be limited to relatively coarse sizes, otherwise lubricants must be used with resultant contamination of the powder. (*3*) Hydriding which gives a friable material easily reduced to a very fine powder by mechanical comminution. The hydride is then either compacted directly and vacuum sintered to remove the hydrogen, or treated *in vacuo* to remove the hydrogen thus producing titanium powder which can be used in the ordinary way. (*4*) Electrolytic processes which give high-purity titanium. Two methods are described to produce high-density parts with good dynamic properties: (*a*) Hot pressing which combines compaction and sintering operations. This is carried out in graphite dies at a temperature between 850 and 1,000°C under a vacuum of about 1 micron. This method is useful for making prototypes or for short runs since it avoids the use of expensive tool steel dies but it does not make a precision product. (*b*) Press forming using tool-steel dies. After pressing, the material is sintered and this can be repeated to increase the density. This method is most useful for fairly large runs since it gives fast production, good utilisation of materials and close dimensional tolerances. The mechanical properties obtainable using titanium and titanium alloys are summarised.

Article by E. P. Weber *Modern Metals* Feb. 1957 48-51

Vacuum Heat Treatment Furnace Makes Titanium Parts Ductile — 242/I

See Abstract No.: 121/III

Preheating of Titanium — 243/I

United Kingdom. Titanium may be annealed and pre-heated for hot working on a commercial scale by conventional heating methods provided certain precautions are taken. A vacuum or an argon atmosphere is necessary only when heating material less than about 0.010 inch thick. In such material slight contamination

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would have a strong effect on mechanical properties because of the high surface-to-volume ratio. Although the absorption of oxygen and nitrogen in titanium above about 800°C is high, the rates of diffusion of these gases through the metal lattice are low so that, provided the heating period is not prolonged, contamination is confined to the surface layers. The scale formed on titanium by heating in air consists essentially of titanium oxides with other minor unidentified constituents present. Underlying the visible scale is a relatively thin layer of metal into which some diffusion has occurred, and this hardened layer must be removed by machining or descaling operations. The effect of nitrogen is less serious than that of oxygen and no nitride has been identified in the scale formed on titanium when it is heated in air at the normal annealing and hot-working temperatures. The chief precaution necessary in pre-heating titanium is to avoid contamination by hydrogen which can be absorbed at the temperatures of 200°C–500°C commonly used for processes such as pressing, stretch forming and drop-hammer forging. Hydrogen has a high rate of absorption in titanium and diffuses rapidly through the metal lattice causing serious embrittlement. The rate of absorption can be reduced by lowering the partial pressure of hydrogen in the furnace atmosphere. A strongly oxidising atmosphere must therefore be maintained in the furnace and contact with hydrogenous materials, including moisture, must be avoided. In order to limit surface oxidation as far as possible, the minimum preheating times consistent with uniform heating should be used. The billet or ingot may be soaked at comparatively low temperatures of 600°C–700°C, which cause only slight contamination, and raised to final temperature immediately prior to forging. Electric furnaces are preferred to oil and gas furnaces but any type can be used. Since the combustion products of oil- and gas-fired furnaces may contain a high proportion of hydrogen, direct impingement of the flame on the metal should be avoided. It is desirable to support titanium in the furnace so as to avoid contact with the furnace refractories and to facilitate air circulation. Induction heating is well suited to titanium since the rapid rate of heating results in short heating cycles which entail only slight contamination. It has been applied successfully in the forging of gas turbine compressor blades and in the hot-heading of titanium bolts and shear pins. Resistance heating is also suitable but very high currents are usually necessary to raise the metal to forging temperature and the method is generally used only for small components. Salt baths and electric blanket furnaces have also been used successfully for preheating titanium.

Article by E. T. Adams *Light Metals* **20**, March 1957 88-90

244/I

The Development of the Iodide Process of Refining Titanium and Zirconium

See Abstract No.: 167/III

245/I

Another Wonder Metal

United States. There is an interesting parallelism in the development of the so-called 'wonder metals'. Titanium, which was discovered in 1789, found no practical application until the development of jet aircraft then production of the metal jumped from 25 tons in 1948 to 14,500 tons in 1956: Zirconium, discovered in 1824, was a laboratory curiosity until recently but production in the next five years will be at the rate of 1,000 tons per annum. This is largely because of its property of passing slow neutrons and thus sustaining chain reactions which now make it the preferred construction material for the core and fuel element cladding of pressurised water reactors including nuclear powered submarines. In addition it has exceptional corrosion resistance (superior to stainless steel); extreme resistance to most acids and alkalies and to the caustic effect of fused metals; a very high melting point; it is as strong as steel but lighter, and fabrication is easy. Zirconium is being used increasingly in steel castings, armour plating, as a cleansing agent for oxides, for furnace linings, crucibles, enamels and porcelain, and is a promising material generally for chemical processing equipment despite its current high price. The best source of the metal is zirconium sand which is found in Florida, North Carolina and California and is also plentiful in Australia; large quantities of the black oxide are found in Brazil and other ores in Colorado. It is difficult to isolate the pure metal as it readily combines with oxygen, hydrogen, silica and carbon. If zirconium is to be used in reactor construction the closely related metal, hafnium, must be removed since it absorbs neutrons at a high rate. The batch reduction process has been used since 1953 but a technological race is developing amongst zirconium producers and continuous processes are being developed. This activity should bring the price down and open commercial markets to the widespread engineering applications of the metal based on its general physical and chemical properties.

Article by Anon. *The Engineer* **203**, 5.7.57 31

246/I

Zirconium

France. A general account of the properties, methods of production and uses of zirconium.

Article by R. Faivre *Energie Nucléaire* **76**, Oct. 1956 32-45

247/I

Zirconium Seeks Markets

United States. By the end of 1957, the capacity of the U.S. zirconium producing industry was expected to reach 6.1 million pounds per year. The chief concerns are Wah Chang, which operates the Bureau of Mines plant, capacity 325,000 pounds, and its own Albany plant, capacity 700,000 pounds; Carborundum, capacity 1.2 mill. pounds; Akron, N.Y., capacity 350,000 pounds; U.S. Industrial Chemical (U.S.I.), capacity 2 mill. pounds; and Columbia National, capacity 1.5 mill. pounds. A combined output of about 4.1 mill. pounds of sponge is forecast for 1958, of which about 3.2 mill. pounds will go to the Atomic Energy Commission, under various contracts, leaving nearly one million pounds available for commercial use. This is not sufficient to develop any large market but is enough to start work on commercial applications. The latter would be

encouraged if the price of the metal would drop to the predicted $3.50 per pound. At the end of 1957 the lowest price for reactor-grade zirconium (low hafnium) was $7.00 per pound in 10,000-pound lots. Commercial grade was $5.00 per pound, again in 10,000-pound lots. The outstanding advantage of zirconium is its corrosion resistance. Carborundum has made heat exchangers, reaction vessels and valves in the hope of stimulating the interest of the chemical and petroleum industries. Wah Chang has a similar project, and Columbia Southern, half owner of Columbia-National, has started a programme to test zirconium under plant conditions. U.S.I. has a one-third interest in Mallory-Sharon Metals, which is scheduled to take over U.S.I.'s zirconium (and titanium) facilities at Ashtabula, and fabricate mill products such as rods, sheets and tubing.

Article by Anon. *Chem. Engng. News* **35**, 16.12.57 30-31

How Tubemakers Work Zircaloy 248/I

United States. Zirconium and zirconium alloys, or zircaloy, are of use as structural materials in nuclear reactors because of their low neutron absorption, good corrosion resistance and high melting temperature. Difficulty is experienced in working the metal, however, because of its rapid work-hardening and tendency to gas absorption. At normal cold working temperatures zircaloy has only one slip plane. Slip occurs along other planes at high temperatures and the metal becomes more ductile, but heated zirconium is highly gas absorbent, to the extent that its properties are seriously impaired. At the Wolverine Tube plant the use of cladding materials to encase the metal and the development of a vacuum annealing furnace have made it possible to produce Zircaloy tubes in standard sizes ranging from 0.25 to 1.00 inch o.d. with wall thicknesses of 0.020 to 0.100 inch. Other sizes can be made to order. The original 1,000-lb. ingots received by the company are first forged on a 1,000-ton hydraulic press to form billets about 8 ft. long and 7¼ inch. dia. These billets are completely jacketed in a cladding material, such as copper, and are then extruded to about 6¾ inch. o.d. and drilled lengthwise. A micro-Kjeldahl analysis is carried out on the turnings to test for nitrogen adsorption. The drilled billet is again completely encased in copper and heated in a rotary gas-fired furnace for 2–4 hours at 1,350–1,500°F before being extruded in a Loewy press under 2,500 psi. The resulting tube is cooled in cold water, pickled to remove the copper sheath, and then reduced to the required size by alternate cycles of cold working, cleaning, annealing and surface treating. Before drawing the tubing is given a phosphate-type coating which serves to hold the lubricant. Heating and cooling is carried out in a special furnace in a maximum pressure of 1μ. The heating zone is a 14-inch. dia. stainless steel retort, 23 ft. long, which is completely enclosed by a gas-fired furnace. A cooling section, also 23 ft. long, is bolted to the crucible on the outside of the furnace. It consists of two concentric mild steel tubes with an annular space for cooling water. The vacuum in both sections is maintained by a National Research Corp. rotary gas ballast pump, a 6-inch. diffusion pump and a small holding pump. The pressure measuring equipment consists of a N.R.C. alphatron and N.R.C. thermocouple gauge. Tubing to be annealed is loaded into a basket which is pushed into the cooling zone by an auxiliary 3 ft. ram. This ram is then removed and the basket is pushed into the heating zone by a 25 ft. ram, on one end of which is a heavy steel cover plate. The plate is bolted to the end of the cooling section to seal the retort. A sleeve in the cover plate permits movement of the ram which pulls the load back into the cooling section at the end of the heating cycle. When the tubing has reached ambient temperature the cover plate is unbolted, the main loading ram is removed and the load is withdrawn by the auxiliary ram. A micro-Kjeldahl test for possible gas pickup is made after each annealing.

Article by J. S. Rodgers *Iron Age* 23.5.57 131-133

Low-Pressure Solubility and Diffusion of Hydrogen in Zirconium 249/I

United States. The mechanical properties of zirconium are adversely affected by the presence of hydrogen, a concentration of 25–100 p.p.m. as normally contained in arc melted sponge is sufficient to cause brittleness and the metal has to be vacuum annealed to 10 p.p.m. or less. Because of the commercial importance of knowledge of the optimum conditions for removal of hydrogen an investigation was made of low-pressure solubilities and diffusion coefficients. Low-hafnium content Zr-O alloys were used with hydrogen obtained from uranium hydride in a heated specimen chamber connected to a vacuum system and the hydrogen was diffused into the apparatus through a palladium tube. An ionisation gauge was used for continuous pressure indication and a McLeod gauge for periodic measurement of absolute hydrogen pressure. This pressure could be regulated to any value in the range 0.1–4μ Hg and maintained at that. Equilibrium solubility was obtained by maintaining constant temperature and pressure for 6 hours after which the zirconium was analysed by the vacuum fusion method for hydrogen content. A duplicate specimen was also prepared for metallographic examination to determine the structure of the metal under equilibrium solubility conditions. It was found that all solubility data follow Sieverts' Law (solubility proportional to square root of pressure), solubility decreases with increasing temperature, and increases with increasing oxygen content at low pressures of hydrogen—apparently because the oxygen in solution expands the lattice to make more interstitial sites available for hydrogen.

To determine the diffusion of hydrogen in zirconium, cylinders of the metal were degassed in vacuum to reduce the hydrogen to 2–3 p.p.m. and then heat-treated in vacuum to dissolve superficial oxygen into the metal as to leave a clean active surface. A specimen cylinder was heated in hydrogen for a predetermined length of time to prepare a suitable hydrogen concentration gradient. After cooling, the cylinder was machined into layers of equal weight which were analysed for hydrogen content by vacuum fusion. Only the inner layers were considered and diffusion coefficients were obtained graphically from the average hydrogen concentrations of each layer. At temperatures below 300°C results were erratic. At temperatures above 600°C this gradient technique could not be used as diffusion was too rapid and an alternative method was used in which the diffusion coefficient was calculated from the rates of evolution of hydrogen from zirconium, containing initially 46 p.p.m., into a vacuum of 10^{-5} to 10^{-6} mm. Hg at various temperatures. The gradient and degassing techniques gave different results and it was concluded that the degassing was not a pure diffusion phenomenon.

Article by M. W. Mallett & W. M. Albrecht *J. Elect. Chem. Soc.* **104**, March 1957 142-146

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A Study of the Hydrogen-Pressure Relationships in the Zirconium-Hydrogen System

United Kingdom. The pressure of hydrogen in equilibrium with a solution of hydrogen in zirconium has been measured for a series of Zr–H alloys at temperatures between 500° and 950°C. The measurements have been used to deduce the form of part of the constitutional diagram for the zirconium-hydrogen system. Hafnium-free zirconium was used for the experiments which were carried out in the pressure range 30–0.3 mm. Hg. The apparatus used comprised a clear silica specimen tube, a liquid air trap, a hydrogen generator and purification tube, a hydrogen reservoir of accurately known volume, a Pirani gauge and a dibutyl phthalate manometer (d = 1.045g./c.c.). When observed with a cathetometer the manometer was capable of measuring pressure with an accuracy of ± 0.02 mm. A Pirani gauge measuring down to 10^{-3}mm. Hg was included to give readings which could be compared with those of other workers. To prevent contamination of the specimen the system was outgassed by heating as much of the apparatus as possible under vacuum after the specimen had been inserted. The part of the silica tube which would be heated in the experiment was maintained at 950°C for at least 24 hours. During this period the specimen was held in an unheated part of the tube and was subsequently moved into working position. The liquid air trap helped to reduce contamination of the specimen by condensable vapours. These precautions limited the pressure rise within the sealed system to about 10^{-3} mm. Hg/hr. in a volume of 25 c.c. when the empty specimen tube was heated to 950°C. Before each experimental run the apparatus was evacuated to 10^{-6} mm. Hg. A known volume of hydrogen was then admitted to the tube and allowed to dissolve in the heated specimen. During each run the specimen tube and the pressure gauge in use were isolated from the remainder of the system by vacuum taps. In calculating the concentration of hydrogen dissolved in the metal it was necessary to allow for the small quantity of gas remaining outside the metal to set up the equilibrium hydrogen pressure within the system. This correction was kept as low as possible by making the internal volume of the closed part of the apparatus small and by keeping as much of the apparatus as possible within a constant-temperature enclosure. The amount of gaseous hydrogen in the heated part of the specimen tube at any given temperature was determined in a separate experiment. It was found that hydrogen produces a marked depression of the α–β transformation in zirconium. A eutectoid point at which the β solid solution is in equilibrium with the α solid solution and a hydrogen-rich phase occurs at 550°C and 32 at .–% hydrogen. The maximum solubility of hydrogen in α–zirconium is 6 at.–%. Small additions of hafnium (approx. 1.5 at .–%) cause a depression of the transformation temperature in zirconium and it is thought that the (α + β) region in the zirconium-hafnium system must pass through a minimum. The results are compared with those of other workers and previous conclusions are reconsidered in the light of the new measurements. A revision of the isothermal section of the constitutional diagram at 750°C is proposed.

Paper by C. E. Ells & A. D. McQuillan *J. Inst. Metals* 85, Nov. 1956 89-96

251/I

The Fundamental Properties of Metals and Powder Metallurgy

United Kingdom. Lattice imperfections and the presence of impurities limit metal properties as known to-day. The need for a study of lattice defects by improved x-ray methods is noted and possible changes in properties as a metal approaches purity are considered. Single crystals of pure iron have been produced by the Westinghouse Research Laboratories which have a breaking strength of nearly a million pounds per square inch. Some have been stressed, without plastic deformation, to 110 tons/sq. inch whereas with ordinary iron the critical stress is 1.78 tons/sq. inch. When titanium and zirconium are prepared by thermal decomposition of the iodide, the oxygen content is as low as 0.01 at .% and the rods obtained are ductile. Vanadium is also ductile if the oxygen and hydrogen contents are below 0.15 at .%, and can be cold worked after initial hot work. Tantalum and niobium bars sintered at 2,500°C *in vacuo* lose their impurities and are ductile when cold. Similarly, molybdenum heated at high temperatures in a vacuum loses grainbound oxygen and becomes ductile at room temperature. In some cases very small additions of various substances are made to a metal to obtain specific properties. For example, sulphur is added to alloys of the Permalloy type to produce a cast metal which can be easily ground at room temperature to provide the powder used in making cores for radio instruments. Additions may also be made to a metal to produce grain coatings which are subsequently attacked by chemicals to embrittle the metal and make it easy to powder. The unusual properties of sintered aluminium powder indicate that it may be considered as a compact of Beilby layers, and its study may lead to further understanding of the Beilby layer. In future research, attempts might be made to produce the Beilby layer in a vacuum or in an atmosphere of hydrogen.

Article by H. W. Greenwood *Metallurgia* October 1956 187-188

252/I

Plating of Tungsten by an Oxidation-Reduction Cycle

United Kingdom. A plating process is described in which a tungsten source is held at a high temperature in an atmosphere of hydrogen and water vapour, with the body to be plated sufficiently close to the tungsten to be heated above 700°C. Tungsten cannot be deposited by conventional plating techniques, and although successful results have been obtained with electrodeposition from fused baths, and deposition by certain vapour phase methods, these techniques require expensive materials which are unpleasant to handle. In addition to providing a coating for other materials a suitable plating method would permit tungsten to be shaped on a former and thus overcome the difficulty of forming and shaping the hard and brittle pure metal. The new method is based on the discovery, made by Langmuir in 1913, that when a tungsten filament was held at high temperatures in a partial vacuum with a vapour pressure of water of about 10^{-4}mm., tungsten powder was deposited on the envelope of the apparatus. In preliminary tests to establish a plating method, a cone of nickel sheet was disposed about a heated tungsten filament in an atmosphere of wet hydrogen. On the parts of the cone heated to temperatures approaching 800°C by radiation from the filament, a coherent and adherent grey film of α–tungsten metal was formed. Modifications led to the development of an apparatus in which the wet hydrogen entered from holes in a supply tube, passed through a tungsten spiral heated to

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2,000°C, and impinged on a nickel tube specimen. The specimen was supported on a rotatable rod and was maintained at 700–800°C by heat from the spiral. With this apparatus a rate of deposition of about 100μ/hr. was obtained. Small specimens coated included a beryllia tube which, when provided with end terminals, formed a small furnace. Shaped parts and foil were made by dissolving out the former on which the metal was deposited. The method gives rapid deposition of adherent coatings under atmospheric pressure using cheap raw materials. The only critical factors are that the water vapour content must be controlled so as to avoid deposition of oxides, the specimen temperature must be high enough to permit the formation of a coherent deposit but low enough to prevent re-oxidation, and the temperature of the tungsten source must be above about 2,000°C. Disadvantages of this apparatus are the need for working in a closed system and the limited life of the tungsten source because of local burn-out. Attempts to make a portable apparatus, for example by using a modified atomic hydrogen welding torch to heat the tungsten electrodes and to clean and heat the specimen, have so far proved unsuccessful. The mechanism of the reaction is not completely understood but two possible explanations considered are: Langmuir's theory that the oxide is reduced by atomic hydrogen, produced at very high temperatures in the vicinity of the tungsten filament; and the theory, based on calculations by Alterthum and Koref, that the reaction

$$WO_2(g) + 2H_2(g) \longrightarrow W(S) + 2H_2O(g)$$

is exothermic so that the equilibrium constant decreases with increasing temperature and makes possible a cyclic oxidation-reduction process. The method appears to be applicable to metals which have a high melting point and a vapour pressure lower than that of at least one of their oxides. Relative ease of oxidation of the metal and reduction of the oxides are also required. Tests have indicated that molybdenum could be deposited in this way.

Paper by M. G. Charlton & G. L. Davis *Trans. Inst. Met. Finishing* **34**, 1957 28-38

Improvements i.o.r.t. Electric Arc Furnaces 253/I

See Abstract No.: 138/III

19 — SCIENCES (other than the above) — 19

Energy Transfers Between Crystals and Adsorbed Dyes I. Desensitisation 254/I

United States. Measurements of the electron affinities of several desensitising dyes indicate that the theory proposed by Tamura and Tutihasi, namely that photoelectrons are trapped in the dye and thus prevented from forming a latent image, is unsatisfactory. The electron affinities of the dyes are found to be less than that of silver bromide and could not therefore 'trap' the photoelectrons. In attempts to measure the sensitisation of photoconductivity in cadmium sulphide the author used a balanced bridge circuit employing two photocells prepared side by side on a glass slip. Application of dye to one cell caused the bridge to go out of balance and tests showed that it was possible to recognise a 1% desensitisation using a wavelength of 5,300 Å. Early results were not reproducible. Contamination of the cadmium sulphide films was found to be due to traces of diffusion pump oil products. Contamination also occurred on leaving the surfaces in the laboratory atmosphere for a week or two. Stringent precautions were therefore taken to avoid contamination, the cells were prepared in an envelope which was then evacuated. Measurements made *in vacuo* were usually repeatable afterwards in air. Slight contamination has a large desensitising effect. Measurements indicate that the electron affinities of the dyes are less than the affinity of the sensitivity specks in a silver halide grain, A dynamic mechanism is suggested as being responsible for desensitising, the theory is that the dye captures most of the electrons formed during exposure and delays their passage to the sensitivity specks so that the latent image is not built up with normal speed and during this time it is unstable. This theory would relate the desensitisation to the reciprocity failure at low intensities, causing a split of the reciprocity failure curve to higher intensities.

Article by R. C. Nelson *J. Opt. Soc. Amer.* **48**, January 1958 1-3

Improvements i.o.r.t. Flexible, Porous, Metallised Sheet Material 255/I

See Abstract No.: 88/III

Radiant Heat Reflecting Clothing 256/I

United Kingdom. It is stated that the numerous patents already in existence, describing heat reflecting clothing depend on the combination of fabrics with sheet metal foil. Flexing and creasing of such material causes deterioration of appearance and possible flaking and peeling off of the metallic layer with loss of heat reflective capacity. According to the present patent articles of clothing are made from metallised flexible sheet material, having a layer of aluminium between 800 Å and 10,000 Å thick. The metal layer may be applied to one or both sides of the material by vacuum deposition. The materials include woven, non-woven or knitted material of asbestos or synthetic polymer, and flame-proofed paper or textile fabric. A heat-reflectivity of 95% is claimed.

R. D. Brace, R. F. Neale & Imperial Chemical Industries Ltd. *Brit. Pat.* 764,335

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I — General Science and Engineering — I

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257/I

Improvements i.o.r.t. Reflective Fabrics

United States. The various known methods of coating fabrics with metal in order to provide infra-red radiation reflecting material for use in protective clothing, etc. suffer from a number of disadvantages: The coated fabric may be less flexible than, or have different draping qualities from, the original cloth; Repeated folding may cause the metal layer to crack and become loosened from the cloth resulting in rapid deterioration when made up, worn, or cleaned; it may be highly porous or may not be continuously reflective. In the method of the invention a thin, highly flexible, solvent- and heat-resistant cured film of an adhesive bonding composition binds the metal surface layer to the fabric base. Basically, a roll of carrier film of offset paper with a thin layer of cellulose acetate on one surface is fed into a vacuum chamber and coated with aluminium by vapour deposition under high vacuum. After leaving the vacuum chamber through a suitable seal the metallised surface of the carrier is coated with a solution of adhesive, the volatile solvent being removed as the composite strip passes through a moderately heated oven. The coated film is then brought into contact with rayon cloth over a heated drum after which the adhesive is heat-cured to a non-thermoplastic state. After cooling, the cellulose acetate carrier is stripped off leaving a highly reflective, visibly continuous and opaque metallic coating on the cloth which retains the flexibility of the original fabric, is solvent-resistant and capable of being dry-cleaned in organic solvents. By keeping the metal layer continuously in full contact with the smooth polished surface of the carrier, and dispensing with the layer of wax the initial reflectivity of the metal layer is maintained. Fire-fighters' 'proximity suits', made by using a pre-formed film of cellulose acetate as the carrier film which was then hot-pressed on to a coarse asbestos cloth, were completely satisfactory under test even after repeated folding and crumpling of the cloth, and without the preliminary soaking in water which is normally necessary with asbestos suits for proximity exposure. Cotton cloth coated by the method of the invention showed no loss of metal coating after dry-cleaning. Such fabric is highly effective as a liner for decorative draperies providing a high degree of protection against sunlight. A wide variety of fabrics, fabric-faced rubber, paper and other flexible fibrous sheet material may be coated by the method, and other metals such as silver, tin, lead, copper and others may be used alone or together with aluminium.

Minnesota Mining & Manfg. Co. *Brit. Pat.* 767,009

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II — Vacuum Apparatus and Auxiliaries — II

Abstract No. and References

20 — PUMPS — 20

High Vacuum Pumps 1/II

See Abstract No.: 2/I

The Early History of the Vacuum Pump 2/II

See Abstract No.: 1/I

The Application of the Centripetal Effect in Air to the Design of a Pump 3/II

See Abstract No.: 4/I

A Centripetal-Pump Effect in Air 4/II

United Kingdom. An instrumental arrangement is described which constitutes a centripeta air-pump. It consists of a hollow cylinder closed at the top and open at the bottom which can be brought into rotation at high speeds about a vertical axis. When a receptacle filled with a heavy oil is lifted until the cylinder is partially immersed, the oil does not wet the cylinder, but an air-gap is maintained between the walls of the cylinder and the oil. It can be seen that air is pumped from the outer atmosphere through this gap into the cylinder. This presupposes that the air must be in a state of stress which includes elastic cross-stresses. It is shown that these stresses result from a stress-strain relation in which the strain is defined in Hencky's logarithmic measure. This confirms Maxwell's theory that air is an elastico-viscous material possessing an elastic shear modulus, and therefore a finite time of relaxation. A rheological equation for air is proposed accordingly.

(*Author*)

Article by M. Reiner *Proc. Roy. Soc.* **240**, May 1957 173-188

Barium Absorption Pumps for High-Vacuum Systems 5/II

See Abstract No.: 15/I

Electronic Ultra-High Vacuum Pump 6/II

United States. An ultra-high Vacuum pump has been developed based on the combined effects of ionisation, excitation and sputtering. A cold-cothode discharge in a magnetic field is employed with no hot filaments nor moving parts. The ultimate vacuum attainable is not yet known but is probably below 2×10^{-10} mm. Hg. A pumping speed of about 10 litres/sec. for air has been recorded at 1×10^{-7} mm. Hg. The pump replaces liquid nitrogen-trapped oil diffusion pumps for pumping microwave tubes.

(*Author*)

Article by L. D. Hall *Rev. Sci. Instrum.* **29**, May 1958 367-370

Improvements i.o.r.t. Vapour Vacuum Pumps 7/II

Liechtenstein. The patent describes a vapour diffusion pump employing the principle of thin-film evaporation for vaporising its working fluid. Advantages claimed are that the design allows efficient evaporation without overheating the fluid, even with a small fluid charge, and that the overall dimensions of the pump are small. The pump has a cylindrical water-jacketed outer casing, which is closed off by top and bottom end sections and has the high vacuum inlet port at one side. From the top end section is suspended inside the casing a double-walled cylindrical structure forming the evaporator a resistance heating element being coiled between the two walls. Inside the evaporator is a rotor mounted on a shaft running axially through the pump, and driven by a motor outside the casing *via* a shaft seal. The rotor vanes just clear the evaporator wall when moving and ensure that the working fluid, fed in through a pipe at the top, spreads out by centrifugal force and forms a thin film running down the hot wall. The resulting supply of vapour is fed through a divergent jet nozzle at the base of the evaporator structure, and is directed downwards into a frusto-conical condenser mounted on the bottom end section of the casing. The pumped gas passes onto a chamber, below this section, to which the fore-vacuum pump is connected. The condensed working fluid also runs into this chamber and drains into an auxiliary pump, driven by the rotor shaft extensions, by means of which it is returned to the evaporator through a pipe outside the casing.

A. Vogt *Brit.Pat.* 781,159

Improvements i.o.r.t. Vacuum Pumps 8/II

United States. The main object of the invention is to provide improved means for supplying the heat required by a vapour diffusion pump to vaporise its working fluid. In normal practice the heater is fixed outside the pump and heats the fluid through the boiler casing. The vapour standpipe and jet nozzles are then heated to their operating temperature largely by condensation of vapour. The present arrangement differs in that the heater is in the form of a coil of resistance wire wound in contact with the standpipe itself, the turns being more numerous at the bottom where the pipe widens out and dips into the pool of fluid in the boiler. The leads to

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the coil are brought out through suitable seals in the pump casing, which may be of metal or glass. Owing to the close contact of heater and fluid it is claimed that warming-up is much more rapid, and that the standpipe and nozzles, being heated directly rather than by condensation, always remain dry in operation. A further feature of the patent, more fully described in Patent Specification No. 767, 277 (See Abstract No. 10/II), is the provision of an auxiliary reservoir into which the fluid charge may be withdrawn out of contact with the heating coil when required.

K. C. D. Hickman *Brit. Pat.* 767,278

9/II

Improvements in High Vacuum Diffusion Pumps

United Kingdom. In a typical vapour diffusion pump, where the upper jet nozzle is formed of a cup-shaped member inverted over the open end of the vapour tube, trouble sometimes arises due to the deposition of small fluid droplets or of condensed vapour on the inside of the nozzle. Such condensation may occur, for example, if the nozzle is at a slightly reduced temperature due to radiant heat loss. The condensate then tends to build up and run through the jet throat down to the rim of the nozzle, from where it drips off on to the next lower nozzle, causing re-evaporation in the wrong direction. To prevent this happening the invention proposes that a raised ridge be provided inside the inverted nozzle member, above the jet throat and of a smaller diameter than the end of the vapour tube. Any accumulation of condensate then drips from this ridge back into the vapour tube instead of running down to the lower rim of the nozzle.

A. A. Aikin & Metropolitan-Vickers Electrical Co. Ltd. *Brit. Pat.* 770,212

10/II

Improvements i.o.r.t. Vacuum Pumps

United States. A disadvantage of conventional vapour diffusion pumps is the necessity for allowing the fluid charge to cool down after operation before air can be admitted. A way of overcoming this difficulty is therefore proposed which provides a means of dissociating the fluid charge from the heating element (the element is preferably a resistance coil wound in contact with the vapour standpipe, as described in Patent Specification No. 767, 278). In one version of the invention there is provided a double walled enclosure between the base of the standpipe and the outer pump casing, forming an auxiliary reservoir opening into the liquid pool in the boiler. If air is admitted to the pump most of the fluid charge is driven into this reservoir and stored there until the forevacuum is reapplied. Since the fluid is largely insulated from the heaters, these may be left energised so as to keep the standpipe prewarmed for quick restarting. A second version has the auxiliary reservoir fixed above the top jet nozzle and connected to the boiler by a tube passing down through the standpipe. In a third design the reservoir is outside the pump and is movable, being connected to the boiler by a flexible tube. Here the fluid may be withdrawn from the boiler at any time by lowering the reservoir manually below boiler level. Another similar arrangement has the boiler base terminating in a length of bellows, which expand when air is admitted and drop the liquid below the level of the heated standpipe. In a further design the complete standpipe assembly is suspended from an evacuated bellows section fixed inside the top of the pump. In this case the admission of air to the pump contracts the bellows, which draws the heated standpipe up out of the liquid pool.

K. C. D. Hickman *Brit. Pat.* 767,277

11/II

Improvements in Diffusion Pumps

United Kingdom. The invention consists of an improved baffle element for mounting in the mouth of a vapour diffusion pump above the top jet nozzle in order to prevent back flow of vapour into the vacuum system. The baffle element comprises an annular member fixed to the cooled wall of the pump and a circular member mounted on the pump axis in a slightly higher plane, an annular gap being left between the two for the pumped gas to pass through. More than one such pair of members may be used in series if required. Each member has its upper and lower surfaces maintained at different temperatures, the upper surface being cooled, for example by water cooling, and the lower surface heated, preferably by an electrical resistance heater. Any vapour molecules striking the upper cold surface are condensed on it, and the surface is sloped in such a way that the condensate drains back into the pump. Molecules from below which strike the lower hot surface are reflected back downwards in the direction of pumping. It is claimed that this arrangement largely suppresses back flow of vapour without appreciably restricting the pumping aperture, and that the end pressure of the pump may also be much improved.

W. Bock *Brit. Pat.* 770,560

12/II

Improvements i.o.r.t. Vapour Vacuum Pumps

United Kingdom. It is common practice to install a cooled baffle between a diffusion pump and a high vacuum system in order to reduce the concentration of pump fluid vapour in the system. The cooling is usually carried out by means of liquid gases or by a mechanical refrigerator, but either method is inconvenient on small pumping systems. The device described in the patent utilises the Peltier, or thermo-electric cooling, effect as a means of refrigeration. The thermojunctions are made of a semi-conducting material such as *p* or *n* type bismuth telluride, with which it is possible to obtain very much greater cooling than with metallic conductors. The baffle structure itself may be of a conventional optical type, composed for instance of annular rings and discs, and is electrically insulated from the walls. It is suspended between two copper terminal rings and joined to each of them by suitable strips or bars of the semi-conductor. The joints at the baffle form the cold junctions and those at the terminal rings the hot junctions, when a current is passed through the circuit in the correct

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direction. The ends of the terminal rings, which are led outside the vacuum system through insulating glands, may be water or air-cooled to dissipate the heat from the hot junctions. By reversing the current the heating and cooling effects are interchanged, and this may be made use of for warming the baffle if required. Since the rate of cooling by such a device is limited, an alternative design shows the baffle as a light spoon-shaped member of very small thermal mass, which is brightly polished to reduce heat gain by radiation.

Edwards High Vacuum Ltd. *Brit. Pat.* 795,895

Improvements i.o.r.t. Vapour Traps for Use with Diffusion Pumps 13/II

United Kingdom. Liquid refrigerant traps for use with vapour diffusion pumps are usually mounted above the mouth of the pump, so that the cold surface is directly exposed to any backstreaming vapour. A disadvantage of this arrangement is that the condensed vapour may freeze and gradually accumulate in the trap, necessitating periodic warming up to return the working fluid to the pump. The present invention provides an auxiliary baffle between pump and trap, consisting of a disc suspended from the cold surface of the trap by a thermally conducting member. The disc is positioned between the cold surface and the pump mouth, so that any vapour migrating backwards strikes it first and condenses on it. The conducting member is so proportioned that the disc runs at a temperature above the freezing point of the pump working fluid, which therefore continually drains back into the pump. If necessary a resistance heating coil may be fixed to the conducting member so that the desired temperature of the disc may be maintained.

Philips Electrical Industries Ltd. *Brit. Pat.* 773,456

On the Factors Determining the Final Vacuum in Vacuum Installations Fitted with Oil Diffusion Pumps 14/II

(Reference only)

Article by R. Haefer & O. Winkler *Vacuum Tech.* 7, Oct. 1956 149-155

Measurement of the Oil Vapour Velocity in a Vapour Pump Nozzle 15/II

Japan. The effect of oil vapour velocity on pumping speed has already been reported by Jaeckel, and work on the measurement of vapour velocity has been carried out by Jaeckel and by Tajima. The author uses a calculation method based on differences in the enthalpy of the vapour. Special consideration is given to the effect of thermal radiation in the measurement of the temperature of the emitted vapour. Although difficulties occur in correcting for the rise in temperature caused by the frictional heat of the high speed gas flow, the measurement of relative vapour speed may be easily made by this method.

(*Author*)

Article by Y. Kondo *J. Japanese Vac. Soc.* 7, July 1957 168-175

The Effects of the Test Dome in the Measurement of the Pumping Speed of a Diffusion Pump 16/II

Japan. In the measurement of the pumping speed on an oil diffusion pump by Dayton's method, the speed measured with a test dome of a larger diameter than the pump mouth shows a smaller value than that obtained with a test dome of the same diameter as the pump mouth. The reason for this is considered to be that the conductance in the former case is smaller than in the latter, when the distance between the pump mouth and the measuring position is smaller than the mouth diameter. The authors have measured the pressure distribution in the axial and radial directions in a test dome, and determined the conditions in the test dome for speed measurements.

(*Authors*)

Article by S. Komiya & T. Ikeda *J. Japanese Vac. Soc.* 7, April 1957 131-147

Rotary Compressors and Vacuum Pumps 17/II

See Abstract No.: 166/II

Improvements i.o.r.t. Rotary Oil Vacuum Pumps 18/II

United Kingdom. Rotary vacuum pumps of the sliding vane type are made with an outer cylindrical stator surrounding a cylindrical rotor, which is mounted eccentrically so as to bear against the stator continuously at one point. At this point locally high temperatures may arise due to friction between the sliding surfaces. The invention therefore provides a pad of material of low coefficient of friction, such as a metal impregnated with polytetrafluorethylene or molybdenum sulphide, set into the stator at the point of contact with the rotor. The spring-loaded vanes may also be constructed of the same materials to reduce friction between them and the surfaces on which they slide. It is claimed that the absence of local hot spots ensured by this means precludes any decomposition of the pump oil which would otherwise worsen the ultimate pressure attainable by the pump.

Electrical & Musical Industries Ltd. *Brit. Pat.* 795,204

Some Hints on the Maintenance of Rotary Vacuum Pumps 19/II

United Kingdom. Regular maintenance of industrial vacuum installations is essential for continuing efficiency.

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Such points as the oil level, V-belt tightness, security of foundation bolts, cleanliness of exhaust valve chamber and mechanism and the clarifier bowl should be checked frequently. Leakages in the gauge line may be rectified by rubbing with castor oil, while those in the oil line call for tightening of the connections. Leaking shaft seals should be re-polished. Leak detection methods vary from the very simple, utilising a soap solution, to the expensive mass spectrometer, used in conjunction with a helium jet. Other methods involve the use of a halogen, acetone paint, or a sealer. Pump seizure may be caused by scoring of the shaft bearings or maladjustment of the V-belt. Excess flow of oil will cause flooding of the cylinder, while dirt may result in the pump knocking. Pipes should be as short and straight as possible and preferably made of copper or steel.

Note by Anon. *Factory Manager* November 1955 15

20/II

Lubrication of Rotary High Vacuum Pumps

United Kingdom. The article deals with methods for preventing contamination of the oil in rotary vacuum pumps by condensible vapours from the vacuum system, and the selection of oils for different duties. The principles of gas ballasting are first described, in which a bleed of air or gas is admitted to the interior of the pump before the compression cycle is complete, so that the partial pressure of any vapour being pumped does not rise high enough to cause condensation at pump temperature. The effect of gas ballasting on the ultimate vacuum and speed at low pressures is discussed. An alternative means for preventing vapour condensation is to use heating coils to raise the temperature of the oil in the pump above the saturation temperature at the discharge pressure; problems of oil oxidation sometimes arise at these elevated temperatures. Another method it to provide auxiliary apparatus which removes volatiles from the oil by distillation or centrifugal clarification. Rotary pump oils are usually mixtures of hydrocarbons having a range of physical properties. Their vapour pressure is typically only 10^{-3} to 10^{-4} mm. Hg. at 80°C., so that their contribution to the ultimate pressure should be negligible. The range of viscosities suitable for general use is about 90–110 cs. at 100°F., but rather higher values are preferred where the oil is heated. The use of low vapour pressure additives to inhibit corrosion and oxidation is often desirable, especially for gas-ballasted pumps or where volatile vapours are being pumped. Additions with a detergent effect tend to be hygroscopic, and their use is not recommended in non gas-ballasted pumps intended to produce the highest vacua. A summary gives details of the types of oil suitable for pumps performing several different duties.

Article by A. C. Smith *Scientific Lubrication* November 1957 12-16

21/II

Oil-Collector to Prevent Loss of Oil in Rotary High-Vacuum Pumps

Germany. In rotary vacuum pumps the exhaust valve must be completely covered with oil to effect the sealing necessary to produce a high vacuum. Consequently, when large air flows are passing through the pump, such as during initial evacuation, drops of oil are unavoidably entrained in the air stream leaving the valve and are lost from the pump unless complicated baffling systems are incorporated. In this design the exhaust valve is surrounded by a housing, inside the main oil reservoir, and extending upwards above the oil level. The housing has a restricted opening at the top for air to escape to the discharge pipe, and has at the bottom a number of small holes so that normally the oil levels inside and outside the housing are equal. However when large air flows occur the excess air pressure in the housing, caused by the restricted opening, ensures that the oil in the reservoir is prevented from entering the holes and reaching the valve. As the flow decreases the pressure in the housing falls and gradually allows oil to return through the holes to perform its normal sealing function. A simple plate baffle is fitted above the restricted opening to precipitate any remaining oil drops entrained in the air stream.

E. Leybold's Nachfolger *Brit. Pat.* 785,168

22/II

Automatic Toepler Pump of Improved Design

United States. A new design of automatic Toepler pump which uses a minimum quantity of mercury and is capable of automatic operation at relatively high speeds is described. A length of large-diameter Pyrex tubing has a gas-inlet tube sealed in at the top which projects downwards to a point about midway in the large tube. A second tube sealed to the large one near the top is the gas-outlet, while a third tube sealed to the side of the large tube rather below the mid-point goes to the pump and leak which operate the " piston ". Both inlet and outlet tubes have all-glass float-type valves with conical, ground plugs and seats. The large tube is divided into two parts by an internal horizontal ring seal tapering downwards at the centre to a dip-tube which reaches almost to the bottom. The lower section of the large tube then functions as the mercury reservoir and contains just sufficient mercury to fill the upper portion to the level of the inlet and outlet valves. Both the upper (piston) chamber and the reservoir are evacuated and the gas to be pumped then admitted through the inlet valve; the outlet valve, being weighted with mercury, remains closed. Air is then admitted through a leak valve to the side tube and so into the reservoir, and forces the mercury up the dip-tube into the piston chamber. Here it soon seals off the bottom of the inlet tube and forces the gas past the outlet valve. Operation is made automatic by means of 3 tungsten wire contacts, No. 1 at the bottom of the reservoir, No. 2 just above the outlet valve and No. 3 in the side tube. When the mercury completely fills the piston chamber it reaches contact No. 2 and closes a relay circuit. An arm carrying a rubber pad closes the leak inlet and the relay starts a backing pump which exhausts the reservoir bringing the mercury down again. When the mercury reaches contact No. 3, the relay opens and the cycle recommences. A pump of this design which has a piston volume of 800 c.c. and an operating cycle of 35 sec. can reduce the pressure in a 2 l system to 0.15% in about 10 min.

Article by G. Urry & W. H. Urry *Rev. Sci. Instrum.* 27, Oct. 1956 819-820

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Improvements i.o.r.t. Liquid-Ring Pumps

23/II

Germany. It is known that the efficiency of liquid-ring pumps can be improved by causing the casing to rotate in the same direction as the impeller in order to reduce friction between the liquid and the casing, but in that case, the bearings required are more complicated and substantially stronger owing to the transverse forces introduced. A new construction is proposed, in which the bearings for the casing and impeller are simplified without losing rigidity. A fixed cylindrical bearings element provides a common bearing for both impeller and casing. The hub bush of the pump casing bears outside this element, and a hole drilled eccentrically through the element accommodates the driven shaft of the impeller. The hub bush is recessed into one side of the casing, and the impeller has a dished shape, so that the blades fit around the outside of the hub bush without touching it; the two parts therefore rotate together in their correct relative positions.

Siemens-Schuckertwerke A. G. *Brit. Pat.* 788,378

Corrosion-Resistant Ejector Steam Connections

24/II

United Kingdom. Vacuum processes involving corrosive chemicals make wide use of steam ejectors constructed from such materials as impervious carbon or stoneware for the source of vacuum. This usually gives a successful vacuum-making system, provided the connecting pipework from the steam supply to the ejectors remains free from corrosion. Mild-steel pipework is not always satisfactory for the final connections and a better arrangement is to terminate it some feet from the ejector and to make the final connection in tubing made from a resistant material such as stainless steel or copper.

Article by Anon. *Brit. Chem. Engng.* **2**, 1957 682

21 — GAUGES — 21

On the Measurement of Very Low Gas and Vapour Pressures

25/II

See Abstract No.: 14/I

The Measurement of Small Vapour Pressures

26/II

See Abstract No.: 149/II

High Vacuum Standards. I. The Design and Construction of a Large Standard McLeod Gauge

27/II

Japan. Recently it has become very important to measure low pressures with great accuracy. However, conventional vacuum gauges may have large errors, usually several tens and sometimes several hundreds per cent. To eliminate the confusion arising from this inaccuracy, the authors started to make a standard gauge and to establish a standard calibration method three years ago. The standard gauge for the calibration of ionisation gauges in the range 10^{-5}–10^{-2} mm. Hg. is a large Mcleod gauge whose volume is 1,000 c.c. and whose capillary diameter is about 1 mm. The calculated error of this gauge is about one per cent at a pressure of 10^{-5} mm. Hg, but the experimentally obtained error is increased to two per cent at 10^{-4} mm. Hg. The reason is considered to be due mainly to the sticking of the mercury thread to clean glass surfaces in a high vacuum.

(*Authors*)

Article by H. Ishii & K. Nakayama *J. Japanese Vac. Soc.* **7**, April 1957 113-120

Hydrogen-Sensitive McLeod Gauge

28/II

United States. This note describes a combination palladium tube and McLeod gauge, which has been used for measuring the hydrogen content of aluminium samples. After five years of daily use, this system is still capable of completing the hydrogen separation in about two minutes, compared with at least twenty minutes required by other types.

Note by C. N. Cochran *Rev. Sci. Instrum.* **29**, Jan. 1958 69-70

Compact Oil McLeod Gauge

29/II

Australia. A gauge conventionally calibrated against a mercury McLeod gauge will give incorrect values if used on a vacuum system without a cold trap. The demand is therefore obvious for a compression manometer to serve for calibrating purposes by maintaining the same vacuum conditions as those existing when the gauge to be calibrated is in use. A McLeod gauge filled with a low-vapour pressure oil which would meet these requirements, is described.

Note by N. A. Florescu *Rev. Sci. Instrum.* **29**, June 1958 527-528

New Device for Measuring Low Gas Pressures

30/II

United States. Describes a simple direct-reading gauge of the compression type. A vertical 17 mm. glass tube is in communication with the system and is sealed at the lower end. This tube contains low vapour pressure oil up to a prescribed level and a tubular plunger which can be lowered by means of a magnet. This plunger is made from a capillary tube, sealed at the upper end, the lower end opening to a bell mouth which is

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normally held above the level of the oil. The air in this capillary thus comes to the pressure of the system, as in a McLeod gauge. To take a reading, the plunger is lowered by the magnet until the mouth is below the surface if the oil and the air trapped in the hollow plunger is compressed. With the plunger lowered to a standard position, the height to which the oil forces itself up into the capillary depends on the gas pressure, and a direct-reading scale is provided on the outside. The instrument is suitable for the pressure range 0.1 to 20 mm. Hg.

Note by R. G. Nester *Rev. Sci. Instrum.* **28**, July 1957 577

31/II

Precise Automatic Manometer Reader

United States. The manual operation of mercury manometers becomes very tedious when many readings are to be taken. The authors have designed an automatic manometer reader and indicator which gives higher accuracy than is usual with a simple visual manometer (±0.2 mm.) or that obtained by measuring pressure using electrical transducers (±1.0). Details of the instrument are given.

Article by J. Farquharson & H. A. Kermicle *Rev. Sci. Instrum.* **28**, May 1957 324-325

32/II

Pulsation Errors in Manometer Gauges

United Kingdom. Manometer gauges of conventional design are subject to errors when the gas-pressure differential is pulsating. Various factors contributing to the production of such errors are analysed and the requirements for their elimination are established. A differential manometer capable of indicating the true time-mean of a pulsating head is described.

(*Chemical Abstracts*)

Article by T. J. Williams *Trans. A.S.M.E.* **78**, 1956 1461-1469

33/II

Description of a Sensitive Micromanometer

United States. A micromanometer is described for the measurement of differential pressure greater than 10^{-4} inch of water with tolerable accuracy. The instrument uses concentric tubes to form the fluid passages and provides for remote indication of the level in the central tube by means of a mirrored float and telescope and scale arrangement. The highest instrument sensitivity obtained by the authors was 3.15×10^{-5} inch of water applied pressure per mm. of scale reading, but with suitable refinement in the optical system, it is felt that greater sensitivities are possible.

(*Authors*)

Article by R. Eichhorn & T. F. Irvine, Jr. *Rev. Sci. Instrum.* **29**, Jan. 1958 23-27

34/II

Brass-Bellows Gauge for Null-Reading Manometers

(Reference only)

Note by M. De Crescente & G. J. Janz *Rev. Sci. Instrum.* **28**, June 1957 468

35/II

A Precision Capsule-Type Pressure Gauge for the Range 0-20 mm. of Mercury

Australia. Describes a gauge in which the sensitive element is a sealed capsule of a type already used in a commercial gauge using mechanical linkages to drive a pointer. In the gauge described, expansion or contraction of the capsule due to changes of pressure in the space surrounding it is transmitted *via* a spigot and ball to a pivoted metal plate. This is pivoted in jewelled bearings and carries a small concave mirror, so that longitudinal motion of the spigot is converted into angular displacement of the axis of the mirror. The end of the containing chamber is a glass plate, so that a lamp-and-scale arrangement can be used. The unit is mounted at one end of a 60 cm. horizontal bar, with a small projector at the other end. The effective optical path is increased to 100 cm. by mounting a plane mirror beside the lamp, the curved scale being alongside the unit. A graticule, formed of six 0.003 inch Nichrome wires, 0.4 mm. apart, is mounted on the projector lens and its image on the scale provides a vernier. The gauge was calibrated against a mercury manometer having arms of 28 mm. bore to reduce capillarity, and isolated from the capsule gauge by an oil manometer which acted as a null-indicator between the two gauges. The calibration was found to be stable except for a zero drift which occurred if the gauge was allowed to remain at an appreciable pressure. This was considered to be the limiting factor for pressures between 10 and 20 mm. Hg. The effective accuracy is believed to be better than 1% at a pressure of 1 mm. and 0.1% at 20 mm. The scale is not quite linear, and the sensitivity is greater than 1 cm. deflection per mm. pressure change throughout.

Article by R. W. Crompton & M. T. Elford *J. Sci. Instrum.* **34**, Oct. 1957 405

36/II

A Vibrating Condenser Manometer

United Kingdom. The principle of the use of a vibrating capacitor to measure small displacements has been applied in an instrument to record cyclic fluctuations of air pressure. A summary is made of the results of simple experiments carried out to investigate factors influencing the amplitude of the signal generated by a vibrating capacitor. A description is given of a simple, robust instrument which has been used to measure fluctuating air pressures and reference is made to the characteristics of the instrument. There is a description of the pressure sensing unit, which uses a bellows; models with ranges 0–1 and 0–3 lb./inch^2 have been made.

Article by J. L. Williams & G. F. Eveson *J. Sci. Instrum.* **35**, March 1958 97

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Modern Developments in the Techniques of Vacuum Pressure Measurements

37/II

United Kingdom. Various pressure measuring techniques developed in the last ten years are reviewed: *The hot cathode ionisation gauge.* This gauge is in effect a triode valve placed in the vacuum system. Positive ions formed by collision between an electron cloud from a hot cathode and gas molecules present in the space between the grid and anode of the triode are collected at the anode. The anode current is a direct measure of the pressure. Unfortunately the anode also acts as an emitter of photo electrons which cannot be distinguished from the positive ions arriving. The ion and electron currents are of the same order of magnitude at a pressure of 10^{-8} mm. Hg, which is the limit of the gauge. A gauge developed by Bayard and Alpert uses a single wire as anode, surrounded by a helical wire grid. The cathode is a hairpin filament placed outside the grid. Because of its small surface area, the anode photocurrent in the gauge is negligible and the range of the gauge is extended to 10^{-10} mm. Hg. Two filaments can be fitted, the second being used when the first burns out. *The cold cathode (Penning) gauge* operates on the principle of the magnetron. An electron cloud is constrained to oscillate forwards and backwards within a cylinder by the action of a magnetic field which prevents the electrons from reaching the anode. The electron flow maintains a high rate of ionisation and the resulting discharge current is proportional to gas pressure between 10^{-3} and 10^{-7} mm. Hg. The performance of the gauge depends to some extent on the gas composition because the ionisation in the discharge causes gas to be cleaned up at the electrodes. Accuracy is maintained as the conductance of the gauge tubulation is not less than 10 litres/sec. *The Micro-Manometer.* The stretched diaphragm differential pressure gauge is a development of the aneroid barometer in which the deflection at the centre of a thin diaphragm is proportional to the pressure difference between the two sides. Cook and Danby have constructed a gauge with a corrugated diaphragm with a depth of corrugated 0.01 inch in a sheet 0.001 inch thick. The diaphragm is part of the capacity in a tuned circuit. Pressure difference in the range of a 0 to 100 microns can be measured to within 2 microns, the calibration being stable to better than 0.5 micron. A similar gauge using bellows in place of the corrugated diaphragm is more easy to construct but only one tenth as sensitive. A gauge using a plane diaphragm and a complex detector circuit is described. Full scale deflections can be 100 or 10 microns. Matheson and Eden have described a gauge in which the diaphragm displacement is measured by finding the change in strain and therefore in electrical resistance of a thin wire stretched between the centre of the diaphragm and an original support. The gauge is linear over a pressure range of 1 mm. Hg with a short period accuracy of 1 micron and 5 microns over a period of days. The sensitivity of all diaphragm micromanometer gauges is independent of gas composition. *The Thermal Conductivity Gauge* depends for its action upon the heat conducted from a hot wire by the gas. The resistance of the wire is measured on a bridge and varies with the wire temperature, which depends upon the loss of heat. Maximum linearity is obtained using a thin wire operated at a constant temperature. The constant temperature bridge needs readjustment before each reading and there is danger of burning out the wire due to over-voltage. A feed back circuit has been designed by von Ubisch which automatically maintains constant wire temperature and provides a direct reading gauge with a useful range up to 10 mm. Hg. Chemical reactions between the gas and the wire can be reduced to negligible proportions by using quartz coated platinum wire.

Paper by J. H. Leck *Proc. Inst. Electronics* 38 2nd Quarter 1955 25-34

Feedback-Controlled Heat Conductivity Gauge for Measuring Pressure in Vacuum Systems

38/II

See Abstract No.: 96/I

A Feedback-Controlled Pirani Gauge

39/II

United Kingdom. A Pirani gauge may be operated with a constant-voltage, or constant-current supply, or with the gauge wire maintained at constant temperature. The author prefers the latter method as giving the greatest useful pressure range and the article describes an electronic feedback circuit which so adjusts the bridge supply current as to maintain this condition automatically. A d.c. system is used, a power valve supplying the bridge current from a 250 V line. The bias voltage of the valve is the difference between 45 V from a battery and the drop across the collector load of a p-n-p transistor whose input current is the out-of-balance current of the bridge. One arm of the bridge is the gauge element, another arm is a variable resistor, used to balance the bridge at ' zero ' pressure. An increase in pressure then causes an increase in the out-of-balance current and a reduction in the grid bias of the valve, so that the supply current increases. Sample results presented show the amplifier to have a gain of about 4,000; the out-of-balance changes between 20 and 40 μA between 0 and 10 mm. Hg, the supply current meanwhile changing between 20 and 100 mA. The calibration is in terms of the supply current. The effect of ambient temperature changes on the transistor characteristics is to some extent compensated by its effect on the gauge filament, and could be reduced by employing two transistors in the input stage. The circuit is claimed to be simpler than the alternative a.c. system and cheaper than a magnetic amplifier of comparable output.

Article by J. H. Leck *J. Sci. Instrum.* 35, March 1958 107

Extended Range Thermal Conductivity Vacuum Gauge

40/II

United States. Two magnetic amplifier circuits for automatically controlling a constant temperature hot-wire vacuum gauge are described. The differential circuit senses temperature variations by means of voltage and current-control coils which respond to the ratio of voltage to current in the hot wire. The bridge circuit senses

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temperature variations by unbalance in a Wheatstone bridge circuit. In either case, the sensing signal is amplified and fed back to the hot wire in such a direction as to very nearly restore the original temperature. Experimental data are given showing good gauge sensitivity up to 50 mm. Circuitry is simple and, since the magnetic amplifier is inherently a stable device with no perishable components, long trouble-free life can be expected.

(*Author*)

Article by A. R. Hamilton *Rev. Sci. Instrum.* **28**, Sep. 1957 693-694

41/II

Wide-Range Thermal Convection Manometer

Argentina. Describes a gauge, operating on the Pirani principle, in which the upper pressure limit is extended by deliberately making use of convection to increase the cooling at pressures where variation in thermal conductivity is small, The gauge consists typically of a tungsten filament of about 300 ohm cold resistance, mounted vertically in a glass tube and surrounded by an inner tubular shield, so that convection causes a current of cold gas to pass the filament, the heated gas returning outside the shield. The filament operates at about 1,000°C., and the speed of response is increased by having a metallic outer wall, although this is not water-cooled. A calibration graph, obtained with a McLeod gauge, shows a useful range from about 5×10^{-3} to about 100 mm. Hg, with maximum sensitivity in the region of 1 mm.

Article by J. A. McMillan & T. Buch *Rev. Sci. Instrum.* **28**, Nov. 1957 881-882

42/II

Wide-Range Thermistor Vacuum Gauge

United States. A new wide range vacuum gauge has been developed for the measurement of pressures from atmospheric pressure to one micron of mercury. The gauge is a heat-conductivity type using a thermistor as a sensitive element. The Pirani principle operates below 10 mm. Hg, but for measuring pressure above 10 mm. Hg the element is placed over the air exit of a blower, and is used as an anemometer.

Note by P. E. Seiden *Rev. Sci. Instrum.* **28**, August 1957 657-658

43/II

High Vacuum Standards. II. Factors Influencing the Calibration of Sub-Standard Ionisation Gauges

Japan. In order to make use of an ionisation gauge as a sub-standard, many factors influencing the sensitivity of the gauge have been investigated in detail. To calibrate a sub-standard gauge against a standard McLeod gauge, it is necessary that deviations in sensitivity are within one per cent in the pressure range from 10^{-4} to about 10^{-3} mm. Hg. Experiments have been carried out under various conditions, *i.e.* changing the values of pressure, the types of gas, electron currents and electrode potentials, for several types and dimensions of gauge. From these experiments the optimum conditions have been obtained for a gauge having a constant sensitivity for pressures up to 3×10^{-3} mm. Hg. However, it was found that the sensitivity tended to decrease slowly during 5 to 25 hours after outgassing by electron bombardment.

(*Authors*)

Article by H. Ishii & K. Nakayama *J. Japanese Vac. Soc.* **7**, July 1957 176-188

44/II

Ionisation Gauges for Measuring Pressures up to the Millimetre Range

United States. By designing an ionisation gauge such that the electron paths are short and well-defined and that the collection efficiency of the ion collector is near unity, one can extend the range of ionisation gauges to pressures of the order of a millimetre of mercury. Several specific tube constructions are proposed and the characteristics of the gauges discussed.

(*Authors*)

Article by G. J. Schulz & A. V. Phelps *Rev. Sci. Instrum.* **28**, Dec. 1957 1051-1054

45/II

Characteristics of the Bayard-Alpert Ionisation Gauge at Pressures above 10^{-5} mm. Hg

United States. Bayard-Alpert ionisation gauges were calibrated in the range from 10^{-5} to about 10^{-1} mm. Hg for various gases. In this pressure range, it is necessary to operate with grid currents below 100 μA in order to minimise the effects of gauge pumping and to prevent space charge from affecting the operation of the gauge. The gauge becomes insensitive to pressure when the ion current reaches 20 to 30% of the electron current. However, linearity is preserved only up to an ion current of about two per cent of the electron current. Some of the reasons for the high pressure behaviour of the Bayard-Alpert gauge is discussed.

(*Author*)

Article by G. J. Schulz *J. Appl Phys.* **28**, Oct. 1957 1149-1152

46/II

On the Abnormal Outgassing in an Ionisation Gauge

Japan. Indication of an ionisation gauge is frequently fluctuated abnormal due to the deposit of oil vapour. In this report I treated the effect of constructive material and of the temperature of envelope. The clean-up of the surface of constructive material depends merely on the temperature of material and is prominent at about 330°C., and if the temperature of envelope is kept about at 100°C., the abnormal out-gassing phenomenon will decrease.

(*Author*)

Article by H. Okamoto *Proc. Jap. Vac. Soc.* **7**, Jan. 1957 71-89

47/II

Improvements i.o.r.t. a Method and an Instrument for Measuring a Vacuum

Liechtenstein. A new type of radioactive ionisation gauge is described which has the advantage that no power supplies are required for its operation. It consists of a cylindrical casing closed by two end plates. Between

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these a shaft is journalled so that it can rotate under the control of a hairspring. The shaft carries an insulating sleeve which supports a movable electrode consisting of two vanes which protrude between two pairs of fixed plates mounted on the interior of the casing. The arrangement is similar to that of an electrostatic voltmeter. The movable electrode is coated with a thin film of a radioactive substance which emits charged particles; strontium 90 is suitable as it is a β-emitter, has a long half-life and does not emit harmful X-rays. If a perfect vacuum existed inside the apparatus the emission of the β-particles would cause the movable electrodes to become positively charged and the stationary electrodes negatively charged. The force of attraction between them would cause the movable electrode to rotate further inside the stationary electrodes until equilibrium was reached under the action of the spring. The potential difference between the electrodes under these conditions is only limited by leakage currents. In the presence of gas at low pressure, the β-particles produce ionisation of the gas and the movement of the charged particles to the electrodes tends to discharge them, equilibrium is reached when the ionisation current is just equal to the rate of arrival of charge at the electrodes due to the β-particles. The reduced potential difference causes the rotation of the movable electrode to a new position. Since the degree of ionisation of the gas depends on its pressure, the position of the vane will also depend on pressure. It is claimed that this manometer is suitable for measuring pressures in the range 10^{-1} to 10^{-4} mm. Hg. If an axial magnetic field is provided to increase the current of ionisation by increasing the effective path length of the β-particles, this can be extended to pressures below 10^{-5} mm. Hg.

Geratebau-Anstalt Bahers
Brit. Pat. 782,534

A Radioactive Ionisation Gauge and its Application to the Measurement of Latent Heat of Vaporisation

48/II

United Kingdom. The object of this investigation was to determine the latent heat of vaporisation of relatively involatile organic substances by using the simplified Clausius-Clapeyron equation which assumes that the vapour behaves as a perfect gas and neglects the specific volume of the solid or liquid. Thus, to determine the latent heat it is necessary to measure the saturation vapour pressure at a given temperature; however, the pressure need not be measured absolutely if a series of results are obtained and a graph of $\log_{10}$ (Kp) against 1/T is plotted, where K is an unknown constant. Previous work in the pressure range 10^{-6} to 10^{-2} mm. Hg has made use of effusion manometers, measurement of the rate of loss of vapour through an orifice by continuous weighing and a capacitance type of diaphragm micromanometer. All these gauges are fragile and difficult to operate. The hot-filament ionisation gauge is unsuitable because the hot filament may cause decomposition of the sample and because the filament may burn out at pressures above 10^{-4} mm. Hg in the presence of oxygen-containing substances. The radioactive ionisation gauge does not suffer from these drawbacks. The Alphatron gauge was used in early investigations but its ionisation chamber cannot be heated above room temperature because the housing of the electrometer valve is bolted to it. Details are given of a new gauge head which was constructed and which used as an ionising source 0.5 mc. of radium 226 sealed in a 1 × ½ inch ' sandwich ' of silver foil. Such a source attains equilibrium in about a month. Special precautions had to be taken with the lead-in wire to the collector as a parasitic voltage was produced if more than one type of glass was used between the wire and earth, as in a graded seal. The collector current was measured by a vibrating reed electrometer, using a null-method in conjunction with a precision d.c. potentiometer. The ionisation current could be measured with a precision of 10^{-15} A, corresponding to a pressure of less than 10^{-6} mm. Hg for most organic vapours. During an experiment the temperature of the gauge head was kept about 20°C higher than the highest sample temperature to be used. Results are given for fourteen organic compounds investigated. Irregular readings were obtained at each extremity of the pressure range; those at the low pressure were believed to be due to a faulty connection for the ' zero reading ', while those at the high pressures were attributed to saturation phenomena in the gauge. Thermal transpiration may also introduce small errors at the lowest pressures. The radioactive ionisation gauge was found to give more accurate results than a Balson-type of effusion manometer as well as being simpler and quicker to use.

Article by J. H. Beynon & G. R. Nicholson
J. Sci. Instrum.
33, Oct. 1956
376-380

Ionisation Gauge Detector for Gas Chromatography

49/II

Canada. In the analysis of gaseous mixtures by the method of gas chromatography it is important to be able to detect the presence of very small amounts of volatile substances in the gases leaving the chromatographic column. A new ionisation detector for this purpose uses a modified R.C.A. 1949 ionisation gauge. The gauge triode valve is run at a grid potential of about +18V which is less than the ionising potential of the helium carrier gas (24.5 V). No plate current flows, therefore, unless impurities are present. Less than 0.5% of the gas flow from the column needs to be leaked through the gauge which, when working at 0.2 mm. mercury pressure, is at least 200 times as sensitive as a thermistor-type thermal conductivity cell. The gauge performance is insensitive to ambient temperature changes and to the changes in pressure and flows rate which occur particularly when a rising column temperature is used.

Letter by S. A. Ryce & W. A. Bryce
Nature
179, 9.3.1957
541

An Improved Ionisation Gauge Control Circuit

50/II

India. Stabilisation of the electron current in an ionisation gauge enables the pressure to be obtained directly from the ion current. An emission stabiliser, with the advantage of a wide range of control, is described. A further advantage is obtained by the use of a grid current amplifier for measurement of the ion current, enabling a wide range of pressure to be obtained on a single logarithmic scale. A 500 s/c oscillator is fed to a variable $-\mu$ pentode, the output being taken to a push-pull amplifier delivering power to the filament of the ionisation tube. The electron current from the grid of the tube is used to control the bias for the variable $-\mu$ valve, in such a manner as to maintain the electron current constant (at 10 mA). Stabilisation to ±1.5% for a power range of 10 : 1 is achieved. Care is taken to avoid instability due to the feedback loop. The ion current is

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Article by P. Hariharan & M. S. Bhalla *J. Sci. Instrum.* 33, Dec. 1956 488-491

measured by passing it to one grid of a double-triode, the other grid being connected to the electrical mid-point of the gauge filament. The double-triode acts as a logarithmic difference amplifier, and is connected to a balanced cathode follower circuit containing the meter. The meter responds to log (i+/i−) where i+ =ion current, and i− =electron current, so that the scale is logarithmic, and residual fluctuations in i− have no effect. The pressure range is 10^{-3} to 10^{-7} mm. Hg. Provision is made for outgassing the electrodes, and for checking the zero and scale of the gauge.

51/II

Rhenium Ion Gauge Filament

Note by L. D. Hall *Rev. Sci. Instrum.* 28, Aug. 1957 653-654

United States. It has been found that a tungsten filament sometimes fails by fracture due to the formation of brittle nitrides when it is heated in an atmosphere containing only a small percentage of ammonia gas. This note reports that a rhenium filament has a much longer life since it does not form stable nitrides. Moreover, its emission is comparable with that of tungsten and its greater resistivity permits the use of thicker, and therefore more rugged, filaments. It is suggested that a rhenium filament might well be used in future as the standard type of filament for an ion gauge.

52/II

Improvements i.o.r.t. Ionisation Manometers

A. H. Beck A. D. Brisbane & Standard Telephones & Cables Ltd. *Brit. Pat.* 771,799

United Kingdom. This patent refers to an improvement to the magnetron ionisation gauge described in British Patent 678,695. The gauge described in the latter consists of a cylindrical cold cathode electrode with field emission surfaces consisting of discs fixed at intervals inside the cathode, each disc having a circular aperture. The anode is a fine wire held taut by a spring coaxial with the cylinder. An axial magnetic field, produced by a solenoid, causes the electrons to follow helicoidal paths about the axis, thus increasing the sensitivity of the gauge. Recent work has shown the necessity for accurately maintaining the anode wire coaxial with the cathode but care must be taken to avoid leakage currents across the insulator supporting the anode wire within the cathode as these interfere with the operation of the gauge. In the improvement described here, the effect of such leakage currents is eliminated by the provision of guard rings to the cathode. These are connected so that they are at the same potential as the anode so that there is no tendency for leakage to occur across the insulators which support the anode wire in the guard rings and any leakage between the guard rings and the cathode does not flow through the load resistance. The performance of the gauge is discussed and reference is made to the use of an alternating magnetic field superimposed on the steady magnetic field; the latter method of operation is described in British Patent 771,800.

53/II

The Behaviour of the Gas Discharge in the Penning Vacuum Meter

H. Moesta *Z. Angew. Phys.* 8, Dec. 1956 598-603

Germany. The saturation currents between the cathode and the anode of a Penning gauge were studied theoretically and measured experimentally as a function of the potential at constant pressure, as a function of the intensity of the magnetic field and of the material of the magnets, and as a function of pressure with constant potential. It is shown that the material of the magnet exerts a strong effect on the discharge and the results show that copper is superior to the German steel V2A. Alnico 400 magnets were used for some of the measurements.

54/II

A Simple Vacuum Leak Detector Using a Radio-Frequency Mass Spectrometer

See Abstract No.: 159/II

55/II

Better Way to Measure Can Vacuum

See Abstract No.: 181/III

56/II

Simple Vacuum Tester for Canned Foods

Article by Anon. *Food Engng.* 29, Feb. 1957 158-161

United States. The apparatus is placed on top of the sealed can so that a gasketted groove forms a vacuum-tight seal with the raised perimeter of the can, leaving a space between the can lid and the testing apparatus. This enclosed space can be evacuated and is provided with a vacuum gauge and an air valve, admitting air to the enclosed space. The vacuum is reduced: the lid returns to its original position: and the air valve closes. The result is a fluctuation of the vacuum gauge about a mean value which differs from the actual vacuum within the can by an amount depending on the construction of the can and the distance between the lid and the air valve. By adjusting the latter the apparatus can be calibrated to indicate the true vacuum inside the can.

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57/II

Development of the Cathode-Ray Oscillograph After 1923

Germany. The story of this instrument is discussed, starting with Gabor's oscillograph which was fitted with two vacuum chambers (the gas-discharge space and the screen container) and evacuated by means of a three-

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stage mercury-diffusion pump backed by a water-jet pump. The greased glass joints of the early models were later replaced by rubber and glass-metal seals. Locks for introducing photographic plates, etc. into the oscillograph developed from three-way stopcocks to U-tubes and rectangular slots. The introduction of thermionic cathodes had to await the invention of the baffle (cooled with liquid air) which allows pressures of 10^{-5}–10^{-6} mm. Hg to be obtained. Early troubles with Apiezon oil are recounted, such as the high price and the frequent damage to the oil due to ingress of air. Recent models are then described which make full use of modern vacuum techniques, such as high pumping speeds allowing attainment of full vacuum after 2–3 min. from closing of the oscillograph, and the automatic isolation of the oil diffusion pump from the fore-pump on opening of the tube. 36 references.

Article by P. Hochhäusler *Elektrotech. Z.* **78A**, August 1957 514-521

The Presentation of Molecular Specimens in the Field Emission Microscope

58/II

(Reference only)

Article by P. Wolf *Z. Angew. Phys.* **6**, Dec. 1954 529-535

The Condensation of Iron and Platinum on Surfaces of Single Tungsten Crystals a Elevated Temperatures in the Field Emission Microscope

59/II

Germany. The surface migration and mobility of foreign atoms on W mono-crystal points were studied by evaporating Fe and Pt in the field electron microscope while the field was switched off. The effects of residual gases on the absorption are reduced by evaporating in a very high vacuum (10^{-9}–10^{-10} mm. Hg) as measured with an Alpert ionisation manometer. The vacuum was produced by an ionisation manometer and the gettering effect of the metals. Growth phenomena were observed with these experiments which are explained by the similarity of the radii of the Pt and the W atoms (1.38 and 1.37 Å resp.). It is assumed that Pd would behave similarly (radius 1.37 Å). The Fe condensates on the various planes vanished with increasing temperature; this is explained by the different energies of surface migration.

Article by K. Neubeck *Z. Naturf.* **11a**, July 1956 587-589

Electron Mirror Microscopy of Magnetic Domains

60/II

United States. Electron mirror microscopy is a rather novel research instrument. It has recently been demonstrated that it can provide micrographs not only of the relief structure of a specimen but also of such purely electrical properties as contact potentials, conductivities, surface charges, and even space charges. This paper deals with the feasibility of electron mirror microscopy for pictorial representation of magnetic properties in general, and of magnetic domain patterns in particular. It is shown that electron mirror microscopy is actually a feasible and independent method for electronically depicting magnetic patterns. The section of the paper dealing with experimental work begins with magnetic specimens of artificial nature, *e.g.* a recorder head, containing one or more linear magnetic dipole stray fields which are controllable. After establishing the basic facts of magnetic contrast formation with this type of specimen, the paper reports on experiments conducted with specimens of barium ferrite and nickel ferrite containing actual magnetic domain patterns. Samples of electron mirror micrographs of domain patterns of these materials are shown and compared with domain patterns of the same specimen areas obtained by the conventional powder technique. The identical nature of the configurations on both micrographs provided final proof of the feasibility of electron mirror microscopy in depicting magnetic patterns. Criteria required to distinguish magnetic patterns from those of electrical or relief origin are derived from the experimental findings and are verified by elementary theoretical considerations about magnetic contrast formation in electron mirror microscopy.

(*Author*)

Article by L. Mayer *J. Appl. Phys.* **28**, Sept. 1957 975-984

Electron Microscopy

61/II

Sweden. The proceedings of the Stockholm Conference on Electron Microscopy are reviewed. Of the 138 communications, about one fifth deal primarily with the development of the electron microscope or the technique of using it. The rest include applications of the instrument to the study of biology, metallography, mineralogy and allied subjects. Electron Diffraction is dealt with in 5 papers.

Article by H. I. Matthews *Nature* **181** 1626-1627

Development History of the Electron Microscope

62/II

United Kingdom. This survey is a revised version of the author's paper read at the Bournemouth convention of the Institute of Physics on May 29, 1953. The early stages of the invention of electrostatic lenses in 1929 (German Patent 690, 809) and of the magnetic microscope of 1931 are referred to in detail and reproductions of early micrographs are given. Assumptions on probable damage to organic or live objects in the electron microscope and attempts at correcting such erroneous beliefs included the argument about the danger of such objects being burnt to cinders or the impossibility of fusing crystals *in vacuo*. The author concludes with a recital of the present achievements in electron microscopy (resolution of 5–6 Å; phase contrast methods; electron interference methods) and a glance into the future (possibilities of development, such as increase in resolution, increased robustness, and reduction of exposure time). 83 references.

Article by D. Gabor *Elektrotech. Z.* **78A**, August 1957 522-530

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63/II

A New Vacuum Plate-Camera of Large Capacity

Australia. In electron optical apparatus it is useful to have a plate-camera of large capacity to avoid admitting air to the system, since a roll-film camera may not have the desired precision. The camera described has a magazine, for storing and feeding the plates, consisting of four large vertical square-threaded screws. The plates are held between the threads, and the four screws are rotated together. The screws are dimensioned so that tolerances in plate size are not important, and so that each plate falls squarely from the bottom of the threads onto a pair of rails. A bar actuated by nylon thread pulls the plate along the rails to a position where a platform lifts it to the exposure position. Centering pins ensure accurate location of the plate, and the platform is concave to the plate, providing some elasticity for the application of pressure. A shutter, which is swung aside for exposure, has the fluorescent screen on the side away from the plate, so that accurate focusing may be obtained. The exposed plate is returned to the rails and carried away by a second bar also actuated by nylon thread. It is dropped into a tray by movement of the rails. The camera is made of $\frac{1}{16}$ inch sheet brass strengthened by brass rods. It slides into the vacuum chamber and the control knobs are coupled to the shafts by a push action. The vacuum chamber is cast in silicon-aluminium. O-ring seals are employed. The camera has a free volume of 4 litres. It is simple and reliable to operate, and will handle 24 plates, size $3\frac{1}{4} \times 4\frac{1}{4}$ inch.

Article by L. N. D. Lucas & P. B. Sewell *J. Sci. Instrum.* **33**, Dec. 1956 465-467

64/II

Observations of Dislocations in Metal by Means of Moiré Patterns on Electron Micrographs

United Kingdom. Electron micrographs of overlapping crystals have shown fringe systems which may be interpreted as moiré patterns arising from the coincidence of the projected planes of atoms in the two overlapping lattices. A technique is described for studying dislocations in metals by observing changes in the moiré pattern. Specimens are prepared by an evaporation technique, in which films of gold in single crystal form are evaporated in predetermined orientation. A second metal is then evaporated on top, such that orientated growth occurs, giving a composite film of two metals each about 200 Å in thickness. An interpretation of the observed patterns is given.

Article by D. W. Pashley, J. W. Menter & G. A. Bassett *Nature* **179**, 13.4.1957 752-755

65/II

Adjustable Apertures for the Electron Diffraction Camera

(Reference only)

Note by J. C. Mills *J. Sci. Instrum.* **34**, Nov. 1957 460

66/II

Electron Diffraction at Electrolytically Polished Surfaces Cleaned by Cathodic Sputtering

See Abstract No.: 101/III

67/II

Electron-Image Recording by Xerography

Australia. In addition to its use for recording images formed by visible light, ultra-violet and x-ray radiation, xerography can also be used for recording images produced in the electron microscope or electron diffraction camera. Using an accelerating voltage of 50 kV electron diffraction patterns have been recorded on vitreous selenium films about 60 microns thick. The principal advantages over the use of sensitised gelatine plates are: (*i*) The selenium plates are free from outgassing, (*ii*) better image resolution may be achieved without sacrificing plate sensitivity and (*iii*) high contrast and wide latitude of exposure may be obtained.

Letter by P. B. Sewell *Nature* **179**, 13.4.1957 773-774

68/II

High Temperature X-Ray Diffraction Camera

United States. Increased demand for materials to operate at high temperatures calls for investigation of properties and of phase transformations of metals and ceramics at such temperatures. It is better to make observations at the high temperature than to rely on quenched specimens. The article describes an x-ray diffraction camera in which the specimen can be examined at 2,000°C in high vacuum, or a somewhat lower maximum temperature if a controlled atmosphere is used. The body of the camera proper is an accurate, vertical cylinder (tolerance $\pm$ 0.00025 inch in 4 inch diameter), having a circumferential beryllium window outside which the film is positioned in a second cylinder fitting over the camera body. The specimen projects into the camera from another cylindrical housing fitting accurately on to the top of the camera body and carrying the motor, gearing and cams for moving the specimen. The camera body is water-cooled, and the various parts are sealed together by O-rings. The lower part of the camera fits on to a vacuum manifold which carries an ion gauge and extends below the table to an internal liquid N_2 trap and a 3-stage 2 inch fractionating pump. The specimen passes through loops in two Ta-ribbon heating strips, which are self-supporting and shielded by Ta-radiation baffles. The x-ray beam passes between the two heaters ($\frac{1}{16}$ inch vertical clearance). A vacuum-tight Vycor window enables an optical pyrometer to be sighted on the specimen, or, alternatively, it can be used to observe the position of the x-ray beam by means of a shutter with a fluorescent coating. The drive to the specimen is transmitted through a bellows motion; it oscillates vertically through about $\frac{1}{4}$ inch once per 8 rotations. For lower temperatures, a thermocouple is used, the leads passing through seals. The construction avoids the use of ceramic insulators, and the film is outside the chamber. Shellac is used to seal on

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the Be-window and Indium gaskets the Vycor windows. A vacuum of 10^{-6} mm. Hg is claimed with the 2 inch diffusion pump. The heater current of up to 250 A is supplied from the mains *via* an a.c. voltage stabiliser, Variac and step-down transformer. This arrangement, together with a pressure regulator for the cooling water, gives negligible temperature fluctuations. A contactor switches off the heating power if either vacuum or cooling water fail. Two x-ray photographs are shown of Ta at 1,730°C.

Article by A. E. Austin, N. A. Richard & C. M. Schwartz *Rev. Sci. Instrum.* **27**, Oct. 1956 860-862

X-Ray Diffraction Studies on Precipitates and Inclusions in Steels Using an Extraction Replica Technique

69/II

United Kingdom. A simple extraction method is described in which precipitates and inclusions in steels can be isolated for identification by x-ray diffraction. The included materials are removed from the metal surface embedded in a thin plastic replica which is formed into a small, compact, cylindrical specimen suitable for powder cameras. The scope of the method is discussed and examples of the application of the method are given. Because the extraction period is only a few minutes the method has several advantages over bulk extraction methods. In addition to the more stable constituents, iron carbide, iron nitrides and metal sulphides have been successfully extracted.

(*Authors*)

Article by G. R. Booker, J. Norbury & A. L. Sutton *Brit. J. Appl. Phys.* **8**, April 1957 155-157

Uses of Carbon Replicas in Electron Microscopy

70/II

United Kingdom. The resistance heating method of vacuum evaporation for the preparation of carbon films and replicas for the electron microscope is compared with earlier methods. The basic replica techniques now in use are described, and applications in a number of fields of research are illustrated. Carbon is the most suitable known material for making support films and replicas, being amorphous, chemically inert and highly electron-transparent. The methods previously used for preparation of these films have all had unsatisfactory features. They have included the glow discharge method, electrical heating of a carbon rod *in vacuo*, and use of a carbon arc. The resistance heating method is superior to these in providing an easily controlled point source with relatively simple apparatus. Two carbon rods are pressed lightly together *in vacuo*, and a heavy current it passed. Intense local heating occurs at the point of contact, causing the carbon to evaporate. Very thin carbon films which are strong, stable and easily visible may be so deposited. Two basic replica techniques are in use. (*a*) The specimen is coated and then dissolved, leaving the carbon. (*b*) A plastic impression is taken and stripped. The negative surface is then coated with carbon and the plastic washed away. This two-stage process permits the preservation of the original specimen, and solid surfaces may be examined. Provided that Formvar is used for the plastic impression, either method can achieve resolutions of 20 Å. Very thin films of carbon should resolve better than 10 Å, given suitable surfaces. Very thin replicas may not provide sufficient contrast, so that where high resolution is not required, replicas may be shadowed or thickened. A pre-shadowed replica is formed by depositing the shadowing material at an angle onto the specimen, and backing with carbon. Less conveniently the shadowing material may be deposited onto the carbon film. By depositing the carbon film at an angle, a ' self-shadowed ' replica is obtained. This must usually be 200–300 Å thick, and some distortion may occur. With the two-stage technique, shadowing must be carried out on the final positive replica. Stereo-electron micrographs may be obtained from metal-shadowed or self-shadowed replicas. Two plates are exposed with the replica tilted at a slightly different angle, to provide a stereo-pair. Carbon replicas find extensive use in the metallurgical field. In general, plastic impressions of etched surfaces are taken and a carbon replica made. In some cases carbon films may be removed directly from the metal. Biological applications include study of yeast cells with a view to investigations of bacteria, examination of some viruses, resolution of micromolecules, *e.g.* proteins; pollen analysis, studies of aquatic organisms and of insects. Carbon replicas have also found many uses in chemical and industrial research. The shape of photographic grains, ultimate size of dense particles, *e.g.* pigments, nature of oxide layers, shape and orientation of clay particles and detail of fibres have all been elucidated using evaporated carbon replicas.

Article by D. E. Bradley *J. Appl. Phys.* **27**, Dec. 1956 1399-1412

Some Carbon Replica Techniques for the Electron Microscopy of Small Specimens and Fibres

71/II

United Kingdom. The difficulties encountered in making replicas of fibres and other small specimens are discussed and three carbon replica methods are described. The first is designed specifically for fibres. The tedious partial embedding techniques which have hitherto been required for such specimens are eliminated. The second is for small specimens which are difficult to handle and range between about 0.5 and 2.0 mm. in size. It has not been possible to use conventional methods with these specimens. The third method is for soft biological specimens such as bacteria. Existing single-stage replica techniques failed with such specimens largely because of the strong chemical reagents required to dissolve the organic materials. All three methods are illustrated by various applications.

(*Author*)

Article by D. E. Bradley *Brit. J Appl Phys.* **8**, April 1957 150-154

Biological Applications of the Carbon Replica Technique

72/II

United Kingdom. Initially the carbon replica technique was confined to the examination of solid surfaces. This article indicates the method used for the preparation of replicas. Some of the applications of the method to the investigation of the minute structures present on the surface of viruses, bacteria, pollen grains and other botanical and zoological specimens are then described.

(*Author*)

Article by D. E. Bradley *Research* **10**, 1957 80-86

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73/II

The Preparation of Electron Microscope Replicas from Rough Porous Surfaces

New Zealand. A two-step collodion-carbon replica process is described for obtaining replicas from rough porous surfaces such as fractured teeth. Specimens are soaked in water and allowed to dry slowly so that a water block immediately below the surface prevents the collodion penetrating too deeply into the pores and ensures that the replica can be stripped. The use of amyl acetate/water interface to float the carbon replica while the primary collodion replica is being dissolved results in less damage to the carbon film than other methods used.

(*Author*)

Article by D. M. Hall *Brit. J. Appl. Phys.* 8, July 1957

74/II

Electron Microscopy of Vulcanised Rubber Using Gelatine First-Stage Replicas

United Kingdom. Water soluble materials have been employed for the replication of rubber surfaces using the two-stage technique. This report is concerned with the use of gelatine. A 20% solution of gelatine is applied to the rubber surface and allowed to dry. The replica is stripped and a carbon film is evaporated as the second stage. The removal of the gelatine is hastened by enzymatic destruction.

Letter by E. H. Andrews & A. Walsh *Nature* 179, 6.4.1957 p729

75/II

Convenient Replication of Textile Surfaces for Electron Microscopy

United States. The method described consists of making a negative replica of the textile surface in polyethylene under moderate heat and pressure, dry stripping a Parlodion positive replica from the polyethylene, shadowing with chromium at 45°, shadowing with carbon perpendicularly and removing the Parlodion by Jaffe's method. The negative replica is prepared by placing a piece of polyethylene 10 or 20 mils thick and about an inch in diameter on the bottom of a stainless steel beaker. The piece of textile is placed on the polyethylene disc and a force of about 10 lb. weight exerted on it by a weight while the beaker is placed in boiling water for about 15 minutes. After cooling, the textile is stripped from the polyethylene and the rest of the method is carried out. It is suggested that the method might be used for other rough surfaces that do not require the highest resolutions.

Letter by W. R. McMillan *J. Appl. Phys.* 28, Feb. 1957 277-278

76/II

A Replica Technique for Particle Surfaces

United Kingdom. Increasing interest in the particle shape and particle size distribution of pigments has led to the application of the electron microscope to the problem. With certain types of pigment the photographs cannot be interpreted accurately because the surface detail of particles cannot be seen, so that clusters of small particles are mistaken for irregular single particles, or, when the pigment has two or more components these cannot be distinguished unless there is a marked difference in particle shape. A replica technique has been developed which is easy to carry out and which reveals this surface detail. The pigment is dispersed in amyl acetate and spread on a clean microscope slide. The particles on the slide are shadowed by the oblique evaporation of gold-palladium and a layer of carbon about 100 Å thick is then evaporated normally on to the slide under vacuum. The film on the slide is cut into small squares for ease in handling and is then stripped off, a high degree of dispersion of the pigment on the slides contributing to ease in stripping. The squares of film are transferred by means of gauze to the surface of a suitable acid to dissolve away the pigment. The concentration and temperature of the acid must be chosen to prevent violent reaction as rapid evolution of gas damages the carbon film. The film is thoroughly washed and then dried. Electron micrographs of replicas prepared in this way clearly show the difference in porosity between calcined zinc sulphide particles and precipitated zinc sulphide particles although the particle size is similar. To distinguish between the separate components of a mixed pigment, only one of the components is dissolved from the film using a leaching agent, *e.g.* a leaching solution consisting of 50 ml. of concentrated hydrochloric acid, 10 ml. of concentrated sulphuric acid and 50 ml. of water, at a temperature of 60–70°C dissolves zinc sulphide completely from lithopone leaving the barium sulphate unaffected. This then appears as black particle among the replicas of the zinc sulphide particles. The technique may be applied generally if suitable leaching agents can be prepared. Carbon films are remarkably resistant to acid attack but a different shadowing metal may be necessary in some cases, *e.g.* platinum has been used when investigating clay samples which were dissolved out in hydrofluoric acid. Reproductions of micrographs show the difference in surface structure of zinc sulphide particles prepared by different methods.

Article by J. Marriott *Brit. J. Appl. Phys.* 7, Oct. 1956 373-374

77/II

Use of B_2O_3 as a Parting Layer for Direct Replicas in Electron Microscopy

(Reference only)

Note by F. W. C. Boswell *Rev. Sci. Instrum.* 28, Sept. 1957 723-724

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Contamination from Copper Specimen Grids During Electron Diffraction

(Reference only)

78/II

Letter by J. J. Comer *J. Appl. Phys.* **28**, July 1957 820

Mass Spectrometers for the Analysis of Inorganic Solids

United Kingdom. A description of two commercial mass spectrometers, one using surface ionisation and the other spark ionisation technique.

79/II

Note by Anon. *Engineer* **204**, 23.8.1957 274

A Simple Vacuum Leak Detector Using a Radio-Frequency Mass Spectrometer

See Abstract No.: 159/II

80/II

Thermal Transpiration in the Mass Spectrometer Inlet System

81/II

United States. The gas entering the Mass Spectrometer leaks through a very small hole into the ionisation chamber from an intermediate pressure vessel (the expansion volume) where the pressure is of the order 10^{-1} mm. Hg. The gas would be in a static condition in both the expansion volume and the inlet tube connecting it to the ionisation chamber but for the effects of thermal transpiration in that part of the inlet tube between the expansion volume and the leak. Transpiration causes a pressure gradient along this inlet tube, so that the pressure at the inlet to the leak is no longer the same as the pressure measured in the expansion volume. It is shown from Knudsen's equation that the pressure gradient always has the same sign as the temperature gradient; the gas density ratio being proportional to the ratio of temperatures at the ends of the tube at high pressures (mean free path small compared to tube diameter) and to the square root of the temperature ratio at low pressures. Considering the sensitivity of the spectrometer, the gas flow through the leak is proportional to the density at the higher pressure side and the sensitivity should be independent of sample pressure. Experiments in which the area near the leak was heated to 195°C showed that the sensitivity decreased continuously with increase of sample pressure above about 1 micron. In the the case of a mixture of gases, the transpiration effect depended on the total pressure of the mixture rather than the partial pressure of the component for which sensitivity was measured. It is concluded that the leak should be placed near the high pressure end of the inlet tube so that any temperature gradient which may arise will be in the low-pressure region.

Article L. A. Dietz *Rev. Sci. Instrum.* **27**, Oct. 1956 817-818

High Sensitivity Mass Spectrometer for Noble Gas Analysis

82/II

United States. The mass spectrometer described operates under ultra-high vacuum conditions and allows gases to be analysed statically, a technique with considerable advantages for argon and the lighter rare gases.

Article by J. H. Reynolds *Rev. Sci. Instrum.* **27**, Nov. 1956 928-934

Application of the Mass Spectrometer to the Study of the Upper Energy States of Molecules

83/II

United States. It is claimed that the poor agreement often found between the energy quantities measured by mass spectrometry, and those obtained by other methods, is due in large part to the interpretation of the experimental data. The conditions necessary for accurate measurement are summarised. It is shown that by making certain assumptions about the probability of ionisation for different processes, ionisation efficiency curves can be interpreted to give the probabilities for electronic transitions from the ground state to the various ionic states of molecules. From these electronic transition probabilities, information can be deduced about the potential energy functions for these upper states. To exploit the method fully, beams of ionising particles with low spreads in energy are necessary. Electron and photon impact are compared, and it is shown that the latter possess many advantages.

(*Author*)

Article by J. D. Morrison *J. Appl. Phys.* **28**, Dec. 1957 1409-1414

Analysis of the Gaseous Contents of Sealed Cathode-Ray Tubes with the Aid of the Omegatron

84/II

Holland. The residual gases in cathode-ray tubes (particularly picture tubes) have been qualitatively analysed with the aid of the omegatron. This is a type of mass spectrograph, small in dimensions, which can be connected to the bulb of a partly-manufactured cathode-ray tube. The residual gases are ionised by a beam of electrons in the omegatron; the ions, acted upon by a constant magnetic field and a radio-frequency electric field, describe spiral paths and impinge upon an ion collector, provided the frequency of the electric field, the magnetic induction and the charge and mass of the ion satisfy the resonance condition. The ion current is displayed as a function of the frequency (*i.e.* of e/M) on a cathode-ray oscilloscope. The principal gases found are hydrocarbons (chiefly methane), argon, CO and CO_2.

(*Author*)

Article by J. Peper *Philips. Tech. Rev.* **19**, No. 7/8 1957/58 218-220

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85/II

Omegatron Analysis of Ions from Heated Salts

United Kingdom. The omegatron has a simple electrode system that can be mounted in a glass envelope and degassed by baking and eddy-current heating and has adequate sensitivity to enable an analysis of low pressure residual gases. Unfortunately, the sensitivity varies with pressure, electron beam current and resolution. It is difficult to distinguish between the several distinct processes which contribute to the performance. For example it is suggested that space charge potential depression causes ion trapping in the electron beam at large electron currents. To reduce the variables concerned, an omegatron is built in which the filamentary cathode is replaced by a heated 0.002 inch diameter tungsten wire coated with a mixture of KBr, NaCl and $SrSO_4$ so that positive ions of K, Na and Sr are emitted. The electrode structure is a hollow 2 cm. cube of copper-nickel alloy and magnetic field strengths are 2.5 to 3.5 kilogauss. The $^{39}K^+$ peak predominates with currents rather larger than those obtained for N_2^+ ion in a conventional omegatron. The background ion collector current is very small. Plotted results show that the peak height varies relatively little with the alternating electric field strength in the upper half of the range used, in contradistinction to conventional tubes. The resolution is found to be inversely proportional to the alternating electric field strength over the mass range 20–200, as suggested by theory. 'Mass harmonic' peaks occur to a serious extent dependent on the method of application of the d.c. ion trapping potentials. The d.c. ion trapping field and the accelerating alternating electric field will both be high at the wire surface but will fall off rapidly with distance from the wire. Experiment shows that the higher the filament current, the higher the alternating field strength to collect the maximum ion current. The radial velocity of the positive ions is very low, so these results are explained on assumption that space charge effects lead to ions experiencing repulsion as they enter beam. This can be off-set at high beam currents by increasing the alternating electric field strength.

Letter by A. G. Edwards *Research* **10**, 1957 288-289

86/II

Vacuum Micro-Balance for the Production of Thin 'Targets' of Given Thickness

Russia. This article is a German translation of the author's original paper in *Pribori i Technika Experimenta* (**1**, 1956, 71–75, in Russian) and describes mechanical and electrical design features of an electro-balance, the vacuum version of which is essential for the determination of the weight of an evaporated film of oxidisable metals, *e.g.* K, Li or Na which when weighed in air, show the weight of the oxide or Hydroxide compounds in addition to that of the metal itself. In the preparation of electron microscope targets the uncoated target is attached to one arm of the balance, weighed in vacuo (10^{-4} mm. Hg), coated and weighed during the coating process. It is stated that subsequent rinsing with argon at 0.1 mm. Hg extends the resistance of the evaporated film against oxidation. The arms of the balance are protected against the metal evaporation by a tantalum shield. A full circuit diagram is given.

Article by W. G. Sykow *Feingerätetech.* **6**, Nov. 1957 487-489

87/II

Weighing under Varying Conditions with a Vacuum Balance

Germany. For some years a vacuum balance has been available which could also be used as a thermo-balance. The mechanism of a normal analytical balance is so arranged that, being housed in a glass cover, it can be subjected to any degree of reduced pressure—up to high vacuum, whilst remaining accessible to the control of an operator. Particular properties of the vacuum balance are described and some indication is made of its wide variety of applications in scientific and technical problems.

(*Author*)

Article by J. F. Cordes *Chem. Ing. Tech.* **30**, 1958 342-346

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88/II

Remote Fuel Fabrication. How it Was Done for EBR-II

United States. The article discusses the practical aspects of the remote-fabrication method for fuel material which has been only partially decontaminated by pyrometallurgical processing. The chief advantages are: Reduction of total fuel inventory; elimination of chemical-recovery plants; reduction of radioactive waste storage. On the other hand, the intense residual radioactivity necessitates remote handling during fabrication of the fuel elements and causes radiation damage to organic materials, such as rubber and lubricating oils, optical glass and electrical insulating materials. In addition, the highly ionised atmosphere produced leads to corona-like discharges at comparatively low voltages. The plant used for the manufacture of the EBR-II fuel pins is described. These are cylindrical rods 0.144 $\pm$0.001 inch in diameter and 14.22 inch long. After rejecting wire fabrication and powder metallurgy, a precision-casting method was developed consisting of a non-mechanical injection-casting process using the pressure of an inert gas (helium or argon) plus the inertial effect of the moving metal to fill the moulds. The metal is melted in a crucible under vacuum by eddy-current heating. A cluster of up to 99 tubular moulds, with the top ends closed, is suspended above the crucible with the open ends pointing downwards. The crucible is raised, immersing the open ends in the liquid and the furnace is simultaneously pressurised with inert gas to several atmospheres. This forces the molten metal into the moulds. The moulds are precision-bore, silica-glass tubes coated on the inside with a colloidal-graphite wash. Precison is made for the remote transfer of the moulds from the furnace, for breaking away the glass and for inspection. Subsequently the elements are assembled in the fuel cans by remote control.

Article by A. S. Shuck *Nucleonics* **15**, Dec. 1957 50-53

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'Clean-Up' of Radioactive Gases Used for Surface Studies

89/II

See Abstract No. : 24/I

Measuring Flux Absolutely with Indium Foils

90/II

United States. Measurements of the absolute disintegration rate of activated indium foil have been made. From these, knowing the thermal neutron absorption cross-section of indium 115, the thermal flux has been computed. This is an alternative to the more usual method of estimation by calibrating an indium foil and particle counter using the neutron flux from a known source or standard pile. A minor modification was made to a proportional counter (P.C.C.-10) in the form of an added cylinder of aluminium which was used to support the foil assembly so as to virtually eliminate back scattering. The only materials responsible for back scattering are then the thin film of Formvar (or collodion) supporting the evaporated indium foil and a ring of aluminium or graphite. In all cases the foils used were commercially pure indium (99.97%), 1cm. square and of thickness varying between 30 $\mu g/cm^2$ and 100 mg/cm^2 for different specimens. For the heavier foils 20 mg/cm^2 –100 mg/cm^2, rolled foil was used; for specimens between 30 $\mu g/cm^2$ and 1 mg/cm^2 indium was evaporated onto a supporting film of collodion whose thickness was about 50 $\mu g/cm^2$. Foils between 1 mg/cm^2 and 100 mg/cm^2 were evaporated onto Formvar films of 2.6 to 3.4 mg/cm^2 mounted on thin aluminium rings. In one case the specimen was prepared by evaporation of indium onto 0.5 mg/cm^2 Pliofilm. All supporting films were mounted on 1½ inch dia. graphite rings, graphite being chosen for its low scattering cross section for indium 116 β-particles and because of its suitability for use in the graphite moderator of the water boiler reactor used for irradiation. All foils used were weighed to determine their thickness down to 1 mg/cm^2 thickness; the assembly was weighed before and after evaporation of the indium; for the thinner foils the thicknesses were determined both by difference weighing and by spectrochemical analysis giving results correct within 2%. The method enabled the final result to be calculated with a total error estimated at ±5%.

Article by M. A. Greenfield R. L. Koontz A. A. Jarrett J. K. Taylor *Nucleonics* 15, March 1957 57-61

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The Control of Mercury Vapour in Laboratories

91/II

United States. The use of large mercury diffusion pumps in a laboratory gives rise to hazards from mercury vapour concentration in the air, especially when maintenance such as filling and cleaning of the pumps is being carried out. The report discusses the methods of control used at the University of California Radiation Laboratory to minimise dangers to health from mercury vapour. Two ultra-violet detectors developed by UCRL are in use : one portable d.c. type, of which a photograph is shown, and one a.c. operated type for continuous monitoring. A special vacuum cleaner, with an activated charcoal filter, is furnished for dealing with visible mercury spilled on floors or benches, and a chemical sealing compound may then be sprayed over the contaminated surfaces if necessary. Exhaust hoods are provided at selected points, and the general ventilation is arranged so that a maximum vapour concentration of 0.1 mg/m^3 of air is not exceeded. However for certain operations, such as filling large pumps, it is recommended that respirators are worn by the operators concerned. The report concludes with a list of safety rules in effect at UCRL, covering routine precautions and procedure to be followed after accidental spillage etc.

Report by C. R. Wintersteen *A.E.C. Rep. No.* UCRL-3714 March 1957

Improved Mercury Vapour Detector

92/II

United States. Many devices for the detection of airborne mercury vapour have been proposed, but usually suffer from the disadvantages of being heavy and cumbersome. This report describes a prototype battery-operated instrument, which works on the usual principle of ultra-violet absorption, but is portable, weighs only 4 pounds, and measures 3 inch × 5 inch × 12 inch. A 6-volt battery, giving an operating life of about 10 hours, provides power for the small blower motor, the amplifier for the output signal from the photocells, and the mercury-arc light source. With further attention to detail it should be possible to reduce power requirements, and therefore prolong battery life, and to reduce the overall weight still further. A circuit diagram of the detector is given, and also photographs of the complete instrument.

Report by T. S. Ely *A.E.C. Rep. No.* WASH-744 October 1957

The Freezing of Water Droplets

93/II

See Abstract No. : 137/I

Physics of Water and Ice

94/II

United Kingdom. A report of a discussion held at the Royal Society on November 14th, 1957, by Prof. J. D Bernal. The report begins by pointing out that earlier theories of the structure of water and ice are inadequate and that practical interest in their properties is increasing, notably in meteorology. A number of British, European and American scientists discussed the results of the latest x-ray, electron and neutron diffraction studies of the structure of ice and interferometric measurements of the expansion coefficient of large single crystals of ice, both ordinary and heavy. These results, and those reported on infra-red absorption in ice, gave little evidence for other than random positioning of the protons along the hydrogen-bonds. Work was reported from Zürich on dielectric relaxation in ice, attributed to hydrogen ions and lattice defects, and from Göttingen on ionic transport, which showed that the apparent velocity of hydrogen ions is greater in ice than in water. It is suggested that this is due to proton transfer over a chain of hydrogen-bonds, and that it is possible (by the introduction of fluorine ions) to create p-n junctions in ice, with protons playing the role of the electrons in

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semiconductors. Work carried out in Amsterdam on x-ray diffraction and infra-red absorption of water suggest that the structure consists of 6 molecular neighbours at 3.0 to 3.4 Å from any given molecule, which differs from earlier views. Work at Imperial College, London, which was presented, discussed the growth of ice crystals, found to be very sensitive to temperature and the freezing of minute supercooled water drops, with and without the presence of nuclei.

Article by J. D. Bernal *Nature* 181, 8.2.1958 380-382

95/II

A Preliminary Study of Some Aspects of a Heavy Water Plant

See Abstract No.: 172/III

96/II

Epoxy Resin as Sealant for High Vacuum Systems

United States. Epoxy sealants consisting of Araldite and 8-10% triethylenetetramine were studied and compared with existing sealing compounds. At operating temperatures of 25–140°C no separation or breakdown of the bond was observed, even when subjected to several successive heatings. The resin partially softens and flows to compensate for expansion of bonded materials. The resin, when bonded for optimum strength, can withstand a force equivalent to 8,000 p.s.i. in compression at room temperature, and 2,000–5,000 p.s.i. in shear as an adhesive. Its vapour pressure is low throughout the operating range, being 1.7–1.8 μa of ion current per Ma of electron current at 25°C as determined by the method of Zabel.

Article by S. S. Stivala & V. L. Denniger *Ind. Engng. Chem.* 49, 1957 1106

97/II

Aluminium and Lithium Greases

See Abstract No.: 178/III

98/II

Lubrication with Molybdenum Disulphide Formed from the Gas Phase

United Kingdon. The lubricating properties of molybdenum disulphide films prepared in a variety of ways are discussed.

Article by F. P. Bowden & G. W. Rowe *Engineer* 204, 8.11.1957 667

99/II

Investigation of the Mechanism of MoS_2 Lubrication in Vacuum

United States. The need for lubricants operating at high temperatures has prompted the study of 'dry lubricants'. These are laminar solids such as graphite or molybdenum disulphide. It would appear that graphite relies for its efficiency on an absorbed vapour layer on the surface and is thus unable to operate effectively under vacuum conditions and the work here reported was carried out to determine whether an adsorbed layer is necessary for efficient lubrication using this material. The apparatus consisted of an evacuated chamber containing a stationary test surface of stainless steel, ground in a standard manner and spaced from a rotating specimen holder by means of three pellets of compressed MoS_2 powder at points near its periphery. The specimen holder was driven *via* a magnetic coupling through the wall of the chamber and the internal magnet was independently supported. No grease was used in the vacuum seals. The chamber was isolated from the diffusion pump by a cold trap and the pressure in the chamber was maintained at about 10^{-6} mm. Hg. After a small correction had been made for the friction at the centre bearing, the coefficient of friction μ between plate and specimen holder was obtained by measuring the angle of lag between driving and driven magnets using stroboscopic illumination. In all cases the value of μ was estimated to be correct within $\pm 5\%$. All measurements were made at a constant peripheral speed of 310 cm/sec. The load on the specimen holder was usually 395 g. although lighter loads gave essentially similar results. The cleaning process for the friction surfaces was standardised and consisted of successive fluxings with organic solvents in a Soxhlet apparatus followed by washing in dilute ammonia, in water and finally heating to 120°C in H_2S. Some samples were also heated under test by an embedded element below the test surface. The original intention has been to investigate lubrication properties as dependent on cleaning procedure, but initial work showed that the characteristic variations were substantially independent of cleaning procedure. It was found that if the MoS_2 surfaces were run until steady friction was observed, the coefficient of friction after further standing was always considerably higher than the equilibrium running value. Results indicate that there is a linear relationship between the initial equilibrium coefficient of friction ($\mu \simeq 0.7$) and the 'stop time' up to periods of three hours ($\mu \simeq 0.3$) and the curve then becomes horizontal ($\mu \simeq 0.35$) for longer rest periods. At any given temperature the slopes of the curves for different samples were the same although there were variations in equilibrium values of μ. The higher values of friction exist for only a short time after resumption of motion and the surfaces show no traces of powdering. Effects of reversal on the orientation of the surface were also studied and friction and stop time curves appear identical for both directions of motion. Much shorter rest periods were required for friction to build up at higher temperatures the largest increase in μ being reached after 30 mins. rest at 50°C. Lower temperatures produced opposite results, at −18°C no increase in μ occurred during the rest period. This variation suggests an evaporation phenomenon, but the characteristic increases in μ do not appear to depend on the

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surface temperature during the waiting period but on the sliding action itself. The authors suggest that an amorphous layer of sulphur is produced by the initial sliding motion, that the evaporation of this layer during standing is consistent with the low vapour pressure of sulphur thus causing a slow increase in μ as the layer evaporates on standing. They further suggest that a fundamental requirement for stable lubrication by laminar solids is that the surface layers of the solid are protected by a film of lower shear strength than the planes of the solid itself, but that the solid surface must provide a low bonding strength for the adsorbed layer.

Article by V. R. Johnson & G. W. Vaughn *J. Appl. Phys.* **27**, Oct. 1956 1173-1179

Graphite and Molybdenum Disulphide

100/II

United Kingdom. This paper compares the lubricating properties of graphite and molybdenum disulphide with particular reference to their use in gas-cooled graphite-moderated reactors. It is stated that the work of Savage on the behaviour of graphite in a vacuum has tended to present a pessimistic view of its lubricating properties in such reactors. Molybdenum disulphide is found to offer advantages in certain conditions but its lubricating action is largely dependent upon the changes in surface activity which accompany comminution. Other workers have found that the lubricity of graphite is not inherent in the crystal structure alone, but depends on adsorbed films. Thus, in a vacuum graphite brushes seize upon a moving copper or graphite base and wear away as fine dust but this wear and accompanying high friction are instantly stopped by some condensable vapour at low pressures. In addition, it is reported that when such a brush is first ' run-in ' for a few minutes in moist air, and then dry nitrogen, negligible wear takes place indefinitely. It is therefore suggested that when graphite is to be used as a lubricant in atmosphere other than air, the surfaces shall first be run in under normal conditions. The graphite will then lubricate efficiently at pressures down to 10^{-6} mm. Hg, if the temperature does not exceed 300°C. Studies of the formation of surface oxide layers in MoS_2 powders are reported; it is believed that the efficiency of MoS_2 as a lubricant is allied to its oxidising properties.

Article by E. R. Braithwaite *Nuclear Engng.* **2**, March 1957 107-110

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Industrial Glass Pipelines

101/II

United Kingdom. Some of the processes used by James A. Jobling and Co. Ltd. in the manfacture of 'Pyrex' industrial glass-ware are described. The basic ingredients of the glass are a pure iron-free sand mixed with ' cullet ', *i.e.* glass broken down for re-melting. These ingredients are fed into furnaces of about 120 tons capacity maintained at a temperature of 1,600°F. Molten glass for use in mould-blown flasks and cylinders is picked up on a blowing tube from access holes in the furnace wall. ' Oldershaw ' columns provide an example of the precision ware which is made by the company under normal glass shop procedure. These columns comprise tubes, 1 or 2 inch in dia., into each of which is fused and sealed a series of thin perforated glass baffle plates incorporating distance pieces. The plates are drilled with a pattern of numerous small holes and must be fused round their edges into the glass column without distortion or closing of the holes. Glass pipeline is produced by a continuous automatic drawing process from a rotating bath which is fed continuously from one side of the glass furnace. As the glass is drawn upwards it passes over a central cone of the required bore size which projects above the level of the molten glass. A flow of hot air passes into the tube as it is formed above the cone, and the glass cools and sets as it is drawn up some 70 ft. in a tower. The continuous drawing process is not suitable for making very large tubes, which may be up to 18 inch dia., and these are mould-blown. Precision glass tubing can be drawn on special drawing machines to within 0.01 mm. of a specified internal dia. and 0.025 mm. of the required outside dia. These machines consist essentially of a horizontal drawbench with a centrally disposed tunnel-heating zone. As the tube is drawn through this zone it is heated to the plastic state and by means of a vacuum the plastic section is drawn on to a precision mandrel suspended in the bore. The drawing speed is adjusted so that the glass sets just as it clears the end of the mandrel. Pipelines are generally built up of 10 ft. lengths of tubing coupled together by mechanical clamps which lock on a short conical section of increased wall thickness at each end of the tubing. The conical section is butt-welded to the end of the tube and this can be accomplished so that, on a normal 10 ft. pipe, the overall length is exact to within $\frac{1}{16}$ inch and the faces at each end are parallel to within 0.088 inch. The welding is carried out on lathes fitted with two headstocks which can be rotated at the same speed, one headstock being fixed and the other movable along the bed. The parts to be joined are mounted in the headstock chucks and as they are rotated they are heated and brought together by an inward movement of the moving headstock. Preliminary heating is effected by gas burners and the glass is then brought quickly to fusion temperature over a small area by means of a special welding process. This process is based on the fact that above a pre-heating temperature of about 350°C the conductivity of borosilicate glass increases as the temperature rises. The preheating gas burners are used as electrodes during the actual fusion operation.

Article by Anon. *Engineer* **203**, 15.3.57 415-416

Photoelectric Effect from Borosilicate Glass

102/II

United States. Values of the photoelectric quantum efficiency of glass for different wavelengths have been determined and data have been obtained on the status of electrons placed on glass surfaces. One of the borosilicate glasses commonly used in discharge tube manufacture and soda glass were studied in vacuum with 2,537 Å photons from a quartz Heraeus mercury arc and with light from a glow discharge operating in pure A and Ne from 10^{-1} to 10 mm. pressure. Clean borosilicate glass in vacuum at 22°C with fields of some 1,000 V/cm. across it yields photoelectric current densities of the order of 10^{-12} A/cm.^{2} for the full light of a 110 V arc focused on 10 cm.^{2} area through quartz optics. This current comes from the 3–4% Na impurity atoms on the surface. Soda glass yields currents in proportion to the density of Na atoms. The normal conductivity of the glass comes from Na^+ ions of some 0.5 V activation energy normally present. It is enhanced 10-fold by Na^+ ions liberated at the surface by light. Na^+ ions next the cathode cannot leave the glass, but electrons enter the glass from the

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metal cathode backing of lower work function to neutralise the ion space charge as it builds up near the cathode. Electrons sprayed onto the glass surface have a mobility of $\sim 5 \times 10^{-5}$ cm.^{2}/volt sec in the glass in contrast to the value of $\sim 1 \times 10^{-7}$ cm.^{2}/volt sec for the Na^+ ions reported by Kraus. Photons above 3,300 Å do not contribute to the conductivity in any fashion. The photoelectric yield for 2,537 Å was obtained directly in absolute magnitude by use of a vacuum bolometer. For the light from glow discharges ⟨⟨1,200 Å, the efficiency was obtained indirectly by comparison with values for Pt and Au. The values obtained are tabulated below.

(*Author*)

Wave-Length A	*Borosilicate glass*	*Soda glass*	*Au*	*Pt*	*Remarks*
2537 A	10^{-4}	6×10^{-4}	10^{-2}	—	Vacuum
»2300 A	10^{-4}	—	—	10^{-2}	Glow discharge
«1250 A	10^{-3}	—	10^{-1}	10^{-1}	0.1 to 10 mm. Hg.

Article by V. K. Rahatgi *J. Appl. Phys.* **28**, Sept. 1957 951-959

103/II

Equilibrium Between Glass and Water Vapour at Bake-Out Temperatures

See Abstract No. : 27/I

104/II

The Adsorption of Water Vapour on Glass Surfaces in Vacuum

Japan. The adsorption of water vapour on lead borosilicate glass in the pressure region of 10^{-6} mm. Hg has been studied using a modified flash filament technique. The amount adsorbed and the kinetics of adsorption could be determined by measuring the variation of pressure after rapid heating and cooling of the sample. The apparatus used in this technique was a Beeck's colorimeter-type cell in which the wire of a resistance thermometer was employed as the heater. The glass sample was crushed to powder finer than 200 mesh and coated on the inner wall of the cell. By this method it was possible to measure the physical and the activated adsorption separately. The results showed that the amount adsorbed by physical adsorption at 10^{-6} mm. Hg and 0–50°C was 0.1–0.4 per cent of a monolayer, and the heat of adsorption was 11 Kcal/mole. Most of the physical adsorption began below 50°C, and the desorption occurred by heating to about 100–150° C or by evacuation. In the case of activated adsorption the activation energy was 9 Kcal/mole for adsorption and 13–40 Kcal/mole for desorption. The residual gas pressure was about 5×10^{-9} mm. Hg, and the variation in pressure was measured by a sensitive (5×10^{-8} mm. Hg/div.) Pirani gauge.

(*Author*)

Article by Y. Tuzi & H. Okamoto *J. Japanese Vac. Soc.* **7**, July 1957 216-230

105/II

Silicones and Their Applications

Reference only)

Article by J. Ames *J. Sci. Instrum.* **35**, Jan. 1958 1

106/II

Bombardment of Plastics in a Glow Discharge

United Kingdom. Experiments have been made on the bombardment of plastics materials such as polyethylene, polystyrene and polytetrafluorethylene in the glow discharge. It was found generally that specimens bombarded with electrons were always damaged whereas specimens bombarded with positive ions were sometimes degraded, for a given set of experimental conditions using an h.t. supply of 5 kV. Material was also sputtered from a surface bombarded with positive ions in air or argon.

Letter by L. Holland *Nature* **181**, 1727-1728

107/II

Vapour Pressure of Plastic Materials

United States. A number of flexible polymeric materials are likely to find use in vacuum systems operated at around 10^{-6} mm. Hg. A method is described for evaluating vapour pressure data for such materials over a range of temperatures, and results are given for a group of them. The method has advantages over high temperature methods, which would involve breakdown, or room-temperature methods which are not applicable if the vapour pressure of the material is less than the base pressure of the vacuum system employed. The sample, of a surface area of more than ten square inches, is placed in a large vacuum vessel which contains an electrically heated oven with an $\frac{1}{2}$ inch orifice in its lid. The oven is baked-out at 450°K for at least four hours and the sample is degassed. Air is then admitted, the sample placed in the oven, and the system pumped down again. Temperature is controlled by a Variac, and measured by a thermocouple and a modified pyrometer attached to the oven lid. Pressure in the oven is indicated by a Vac. Tube Products type 6578 quick-reading ionisation gauge tube, and that in the outer vessel by a Veeco RG-75 non-burnout ionisation gauge tube. Both are operated and controlled by a Veeco RG-3 ionisation gauge control. The pressure in the larger vessel is maintained at a tenth, or less, of the oven pressure, when the vapour pressure is given by the difference in the two pressures.

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Readings are taken at a dozen temperatures in the range 300–450°K, and a new run commenced to ensure reduplication. The results of at least half a dozen runs are given in a table. The results obtained are used to plot vapour pressure *vs.* reciprocal temperature curves. Below about 1 mm. Hg the relation log $P=A-B/T$ holds, and the best straight line is drawn through the point. Since the gauge tubes were calibrated for use with nitrogen, the values of vapour pressure, found by extrapolation and shown in the above mentioned table, are of ' equivalent nitrogen pressure '. The elastomers butyl rubber, Hycar rubber and elastic vinyl have noticeable high vapour pressures. Plasticiser content is likely to be a major factor in these cases. In view of the results, it is likely that some of the polymers tested will find wider application as gasket materials, dielectrics, low friction surfaces and insulators for vacuum systems.

Article by N. Jensen *J. Appl. Phys.* **27**, Dec. 1956 1460-1462

Vacuum Venting of Moulds

108/II

United States. A problem frequently met with in the injection moulding of plastic articles is that of trapped air in mould cavities. Compression of the trapped air by the inflowing melt causes burns and faults at these points in the moulded article. Design shapes are considerably limited by this problem, which is difficult and costly to correct. An inexpensive and effective technique for exhausting the air before injection is described. The method is simple, of general applicability and enables perfect pieces of a wide range of design to be moulded. An example of a two-cavity vacuum-vented mould is illustrated. A small cylindrical container with snap-on cover, which could not be successfully moulded without vacuum-venting, is produced without elaborate equipment. A $\frac{1}{4}$ or $\frac{1}{2}$ h.p. pump, displacing about 5 cu. ft. and drawing down to $\frac{1}{4}$ inch Hg, exhausts the mould *via* a solenoid-operated air valve a few seconds before the electrically-timed injection stroke. Venting is accomplished by machining shallow recesses in the mould face between the runners and two air channels. These form orifices when the mould is closed. The recesses may be as wide as it is convenient, but not so deep or short that plastic material reaches the vacuum line before solidifying. By venting from the runners, material flashed into the orifices does not require subsequent trimming. Orifice size must also be consistent with sufficiently rapid exhaustion. The mould faces should be parallel with good finish, and ejector pins fitted with ' O '-rings or other good seals to ensure air-tightness. The cost of the finished mould and connection should not exceed an increase of 10% above the conventional.

Article by G. S. Bohannon *Mod. Plastics* **34**, Dec. 1956 162-163

Refractories and Mould Materials for Vacuum Melting and Casting

109/II

United States. Experimental work has indicated that the magnesia or alumina refractories commonly used for lining melting furnaces are suitable for vacuum melting the iron-, nickel-, and cobalt-base alloys of interest to the investment-casting industry. If the operating temperatures exceed about 1,590°C, zirconia may give better results than magnesia or alumina because of its greater thermal stability. The partial pressure of gaseous silicon monoxide over silica has been found to be 2.1 mm. at 1,300°C and this indicates the likelihood of reaction between silica moulds and elements such as carbon, aluminium or titanium when alloys containing these elements are cast into silica moulds in vacuum. Studies on the deoxidation of pure iron, nickel and cobalt in vacuum indicate equilibrium between the melt and a magnesia crucible at about 0.005% oxygen for iron, 0.003% oxygen for nickel and 0.001% oxygen for cobalt. With iron melts made in alumina and zirconia crucibles the equilibrium contents were 0.005 and 0.007% respectively. Thus there appears to be little practical difference in the oxygen levels that can be attained in magnesia, alumina and zirconia crucibles under normal vacuum-melting conditions. The level of oxygen contamination from high purity crucibles is not excessive for commercial applications. In investment moulds for vacuum casting the time available for reaction is short and silica investment moulds appear adequate for most metals. Trouble may be caused however by any reaction which results in gas formation and by the volatilisation of refractory components in contact with hot molten metal. Moulds bonded with ethyl silicate have shown satisfactory behaviour during vacuum casting but violent gassing occurred when molten metal was poured into moulds bonded with phosphate although the moulds were heated to 1,010°C before casting. Where reactions are likely between the alloy and the silica investment mould, precoatings made with alumina, zircon or zirconia may be useful. In some cases lowering the mould temperature prior to castings, for example from the heating temperature of 1,010°C to 705°C, reduced the tendency for gas formation. Greater permeability of moulds is an advantage in vacuum casting if gassing is likely as the gas can then escape without being forced into the metal. Shell-moulding techniques such as the ' Mercast ' or ' Glascast ' processes appear particularly advantageous in this respect. In the discussion following the presentation of the paper it was said that centrifugal casting might overcome the effects of gases in moulds of low permeability, but this would complicate the vacuum casting process and would not be suitable for certain shapes. An advantage of combined vacuum melting and casting is the elimination of hydrogen pick-up. If the alloys contain reactive elements such as aluminium and titanium, loss of these elements by oxidaton is prevented and in addition improved ' castability ' is achieved. Some casting was carried out under inert gas and when casting cobalt-base alloys containing 0.5% carbon into silica investment moulds, excessive gas formation and undesirable metal penetration into the mould surfaces were eliminated by introducing argon into the vacuum chamber just prior to making the cast.

Paper by H. O. McIntire *Foundry Tr. J.* 7.11.57

Coating a Carbonaceous Surface with Rhenium

110/II

See Abstract No. : 218/I

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111/II

Changes in the Structure of Carbon During Oxidation

(Reference only)

Letter by J. D. Watt & R. E. Franklin *Nature* **180**, 30.11.1957 1190-1191

112/II

Indium Seals for Dismountable Vacuum Systems

See Abstract No. : 143/II

28 — APPARATUS COMPONENTS — 28

113/II

Copper Foil Trap of Improved Form

United States. Describes a modified form of the liquid N_2-cooled corrugated copper trap introduced by Alpert, to reduce backstreaming. Thin corrugated O.F.H.C. copper sheet was packed into a 6-inch length of the 41 mm. Pyrex pumping tube. The inside of this tube was silvered and the section containing the copper shielded by two larger Pyrex tubes sealed to the main tube. The annular space immediately surrounding the silvered portion of the main tube could then be filled with liquid N_2, itself being surrounded by the annular space between the two larger tubes, the whole forming a small integral Dewar flask, very economical in refrigerant. It has been found that such copper traps are very effective in reducing backstreaming even when not refrigerated. Grade-A nickel sheet, specially treated, has been successfully tried as an alternative to copper.

Article by E. Buehler *Rev. Sci. Instrum.* **28**, June 1957 453-460

114/II

Condensable Vapour Extraction Apparatus

United States. The initial pump-down period of large tanks can be approximately halved by employing cold traps to remove the condensable vapours. After the initial pump-down the required pressure can be maintained by the pumping system without the aid of the traps but if they remain in the system the supply of coolant must be maintained to prevent the evaporation of the frozen vapours. This is wasteful, especially if the tank is to remain pumped down for a prolonged period. The invention described relates to a retractable moisture trap used to condense vapours in the space to be evacuated during the pump-down period, and thereafter to be sealed off or withdrawn from the evacuated space. The apparatus consists of a cylindrical vessel which contains the coolant and which is supported in a housing which may be attached to a flange on the side of the vessel to be evacuated. The coolant vessel is provided with two concentric tubes one of which serves to permit the escape of coolant vapour and the other for the displacement of the coolant when the first tube is closed. Means are provided for withdrawing the coolant vessel and its drip-tray until a plate suspended from its base makes a vacuum-tight seal against a rubber gasket mounted on the base of the housing which protrudes some way into the vacuum vessel. This isolates the coolant vessel and the frozen vapours from the evacuated vessel. The space above the seal is evacuated by a by-pass line to reduce the chances of leakage. An alternative arrangement permits the coolant vessel to be retracted past a high-vacuum valve; if this is then closed, the cold trap may be completely detached from the system.

E. O. Lawrence & U.S. Atomic Energy Comm. *U.S. Pat.* 2,737,779

115/II

Measurement and Analysis of the Hold-Up of Gas Mixtures by Charcoal Absorption Traps

United States. This report is concerned with the hold-up behaviour of gaseous fission products in charcoal traps; such traps have been used to reduce the activity of these products to a safe level before venting to the atmosphere. This is achieved by holding up the products until the activity has decayed to a safe level. Moreover, in the case of a sudden burst of activity they reduce the rate of activity in the volume by releasing it slowly over a long period of time. In the investigation 10-year krypton was used, so that the decay was negligible, and either nitrogen or helium was used to sweep the radiokrypton through the trap which could be maintained at various temperatures. The concentration of radiokrypton in the effluent gas, and therefore its partial pressure, was determined by measuring its activity by means of an end-window Geiger tube. Graphs are given of the relative effluent activity against time for a one-trap and a two-trap system, using nitrogen purge gas, at +16°C, +5°C, −51°C, and −110°C. These show that the time for activity to break through, the time for activity to reach peak and the time for the last appearance of activity, all increase with decreasing temperature. The maximum concentration of radiokrypton in the effluent gas is lower for lower temperatures. With two traps in series the time to peak is twice as long and the break-through time is from two to three times as long as for one trap; the maximum concentration is about half as great. Similar graphs using helium as the purge gas show that the hold-up times are greater than for nitrogen and the maximum concentration is less. This is because charcoal adsorbs helium to a less extent than it does nitrogen. An analytical expression is given which can be used to predict hold-up curves for gases when the trap geometry and the linear adsorption isotherm for the gas mixture in charcoal are known. This enables as optimised trap to be designed for any purpose. For example, if a low maximum concentration is needed, a short wide-diameter trap operating at a low temperature would be best.

Report by W. E. Browning & C. C. Bolta *Oak Ridge National Laboratory Lab.* ORNL-2116

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A Small Mercury Cut-Off Withstanding Large Pressure Difference

116/II

United Kingdom. A mercury cut-off is described which is vacuum-tight, is capable of withstanding a pressure difference between atmospheric pressure and 10^{-3} mm. Hg, is small and has a minimal hold-up of gas. The tube leading to the high vacuum side is closed by a sintered glass disc below which is a mercury reservoir. The level of the mercury can be raised in order to immerse the sintered disc by operating a screw clip which squeezes a length of pressure tubing forming part of the reservoir; this cuts off the two sides of the system from each other.

Letter by H. Block *Nature* **178**, 8.12.56 1307-1308

Alloy-Filled Cut-Off for High Temperatures

117/II

Canada. A vacuum cut-off using low-melting point alloys such as Wood's metal is described. This device can obviously be used only above the melting point of the alloy, but has the advantage over the mercury cut-off of lower vapour pressure. It has been used with success at temperatures up to 200°C.

Note by S. Toby & K. O. Kutschke *Rev. Sci. Instrum.* **28**, June 1957 470-471

A High-Vacuum Valve

118/II

Rumania. A glass tube connecting to one part of the apparatus is sealed into the upper end of a larger one which has a side arm forming the other connection. To the lower end of this inner tube is sealed a short length of Kovar tubing, terminating in a tantalum collar. To close the valve, a Pyrex cup containing metallic tin is raised by means of a solenoid acting on an iron cylinder fixed below the Pyrex cup. The tin is first melted by an h.f. coil surrounding the valve and the cup is raised until the tantalum collar at the end of the Kovar tube is immersed in the molten tin. The tin is then allowed to solidify, when a vacuum-tight seal is obtained, provided that the outer surface of the Kovar shall first have been tinned, the tantalum collar retaining a little tin so as to preserve the tinning of the Kover. With this valve, it was possible to maintain a pressure of 5×10^{-8}mm. Hg (as measured on a Bayard-Alpert gauge), no leakage from atmospheric pressure being noticed during 50 hr.

Note by L. Blanaru *J. Sci. Instrum.* **35**, May 1958 184

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Ultra-High Vacuum Technique

119/II

Japan. Metal valves suitable for ultra-high vacuum have been made according to the original design of Alpert, and examined using a helium leak detector. Inverted-type ionisation gauges, or Bayard-Alpert gauges, have also been made and their characteristics measured in detail. Using the above components, an ultra-high vacuum system has been constructed. A vacuum of 10^{-10} mm. Hg could be readily obtained after two or three hours baking and outgassing of the ionisation gauge. However, the highest vacuum achieved was not so good as reported by Alpert because of incomplete outgassing of the system.

(Author)

Article by H. Arata & Z. Oda *J. Japanese Vac. Soc.* **7**, July 1957 197-215

New Ultra-High Vacuum Valve

120/II

See Abstract No.: 32/I

Improvements r.t. Manually Operated Diaphragm-Type Valves

121/II

United Kingdom. The valve consists of two main sections: A body portion containing the inlet and outlet connections, and a cover plate screwed to it. Between the two is a falt flexible diaphragm of rubber or plastic material, held clamped round its periphery. The inlet and outlet connections communicate with openings inside and outside an annular valve seating in the body underneath the diaphragm. Closing the valve is effected by tightening a screw in the cover portion, which causes a self-aligning pressure pad inside the cover to force the diaphragm against the seating. A spiral spring under the diaphragm ensures that it lifts clear of the seating when the screw is released to open the valve.

J. Blears & Metropolitan-Vickers Electrical Co. Ltd. *Brit. Pat.* 759,492

Magnetically Operated Needle Valve

122/II

Australia. A solenoid operated needle valve is described which gives smooth and rapid control of gas flow rates over a wide range from a cut-off value of approximately 10^{-4} micron-litre sec^{-1}. In conjunction with a suitable pressure transducer the valve may be used for the automatic control of pressure in vacuum systems.

(Author)

Article by J. O. Cope *Rev. Sci. Instrum.* **29**, March 1958 232-234

Controlled Capillary Gas Leak

123/II

(Reference only)

Article by R. K. Smither *Rev. Sci. Instrum.* **27**, Nov. 1956 964-965

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124/II

Article by S. Kobayshi & K. Yada *J. Japanese Vac. Soc.* **7**, July 1957 189-196

A Standard Leak Utilising Gas Permeation

Japan. A small standard leak has been constructed utilising the permeation of a probe gas through silicone rubber sheet. As the leak rate is determined by the area and thickness of the rubber sheet a leak smaller than 1×10^{-7} lit. mm./sec. can be made easily and reliably.

(*Authors*)

125/II

Article by S. A. Gordon *Rev. Sci. Instrum.* **29**, June 1958 501-504

Construction of Small Fixed Leaks of Predictable Throughput

United States. A method is described by which glass capillary leaks with extremely small flow rates can be easily and rapidly produced. The predictability of these leak rates is determined by the resolution of the optical system used in measurement of bore diameter. Data are given which will allow construction of such leaks. A manifold of leaks is described which will introduce gas into a high vacuum system at several different fixed leak rates. It contains no moving parts or high vapour pressure material on the high-vacuum side, yet the leak rates can be manipulated over a wide range. The characteristic features of these leaks are shown to be reasonable in the light of well established theory.

(*Author*)

126/II

Note by C. D'Amico & H. D. Hagstrum *Rev. Sci. Instrum.* **28**, Jan. 1957 60

Improvements in the Use of the Porcelain Rod Gas Leak

United States. A porcelain rod gas leak for admitting gas to a vacuum system has been described previously. The improvement consists in the use of two mercury column cut-offs, which consist of vertical glass tubes, a stainless steel bellows being sealed to the lower end of each. When the bellows is compressed by a screw, mercury is forced upwards to provide a cut-off. A small tube carrying the porcelain rod is sealed to the side of one of the vertical glass tubes referred to above, the end carrying the porcelain being sealed through the wall of the other. The gas reservoir is connected to this second vertical glass tube, so that gas can leak through the porcelain and so into the first vertical tube, which leads to the vacuum system. By raising the mercury column in the first tube, the vacuum system is isolated from the leak, so that the vacuum can be broken without affecting the gas supply. On the other hand, if the mercury is raised in the second tube, it surrounds the porcelain rod and so isolates the gas reservoir from the rest of the system.

127/II

Report by R. Millican *AEC Rep. No. K Y-166* Feb. 1956 1-5

Low Flow Variable Leak

United States. The design is described of a variable leak for a mass spectrometer ion source to provide smooth control of gas flow over the range from 20 Std. c.c./min. to less than 0.002 Std. c.c./min. (1 Std. c.c./min. =12.67 lusec) with an inlet pressure of 1 atmosphere and outlet pressure of less than 200 micron, to be readily maintainable and cleanable through ease of assembly and interchange of components, to be constructed of materials resistent to uranium hexafluoride, and to be able to detect downstream a change in the gas concentration upstream in less than 0.8 min. at an air flow rate of 0.05 Std. c.c./min. at an inlet pressure of 1 atmosphere. Full details of the leak are given in a diagram.

128/II

Note by W. D. Edwards *J. Sci. Instrum.* **35**, March 1958 111

An Insulated Vacuum Lead-In Using an O-Ring

(Reference only)

129/II

Counterbalanced Bellows in Vacuum Systems

See Abstract No.: **33**/I

130/II

Article by Anon. *Lab. Practice* **6**, August 1957 465-470

Ground Glass Joints in the Laboratory

A review.

131/II

Note by G. W. Gore *J. Sci. Instrum.* **34**, Nov. 1957 459

A Simple Glass Sliding Joint for Vacuum Work

(Reference only)

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Vacuum-Tight Flange Joint Between High-Vacuum Pipes

132/II

Germany. This patent describes a means for effecting a high-vacuum-tight joint between pipe flanges which avoids the danger of stressing the rubber sealing ring beyond its elastic limit. This is achieved without the necessity for precision machining of the parts of the joint. The joint consists of an annular distance ring which is provided with a cylindrical centering projection which extends into the bore of one of the pipes and serves to locate the distance ring coaxially with the pipes. The outer circumference of the ring has a groove with a concave cross-section in which is mounted the sealing ring. When unstressed, this has a height somewhat greater than the height of the ring. When the joint is assembled by clamping the pipe flanges together the ring is compressed in the axial direction of the pipes but can expand freely in the radial direction of the ring itself. If the height of the distance ring and the thickness of the rubber sealing ring are conveniently chosen, it is impossible to stress the rubber beyond its elastic limit. An alternative form of the joint consists of a ring with an external rib of rectangular cross-section on which is moulded a not very resilient sealing material, such as a high-polymer synthetic material.

M. Dunkel & E. Leybold's Nachfolger *Brit. Pat.* 783,397

Step-Type Demountable Metal Vacuum Joint

133/II

United States. Demountable all-metal vacuum joints are reported which employ oxygen-free high-conductivity copper as the gasket material between stainless steel flanges. The step-type assembly is easier to machine and tolerances are wider than in the knife-edge type of seal. The seals were tested over repeated cycling to 450°C under ultra-high vacuum conditions.

Note by W. J. Lange & D. Alpert *Rev. Sci. Instrum.* **28**, Sept. 1957 726

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Demountable Metal Vacuum Seal

134/II

United States. Standard stainless steel pipe fittings have been adapted to make easily demountable vacuum-tight joints using metal gaskets. Aluminium has been used as the gasket material with success, but rubber, Teflon, copper, silver or gold should work equally well.

Note by T. Foote & D. B. Harrington *Rev. Sci. Instrum.* **28**, July 1957 585-586

Improvements r.t. Vacuum Hose Couplings

135/II

United Kingdom. Vacuum hose coupling heads of the 'Clayton' type, as used in railway braking systems, are usually made with one end tubular, for connection to a flexible hose, and the other end flanged to mate with a similar flange on the other head of the coupling. The flanges carry suitable lugs which keep the coupling in engagement, and are provided with grooves into which soft sealing rings are fitted. Each coupling head may either be made in one piece, with the groove machined afterwards, or fabricated in such a way that the groove is formed between adjacent shoulders of separate pieces. The present design is intended to cheapen manufacture by using two die-cast parts : a tubular body portion and a flanged portion. The flanged portion is taken hot from the mould, and shrunk into position over the cold body portion. Stepped shoulders cast into the two portions abut in such a way as to define a groove for the sealing ring, which requires no further machining.

L. P. Candor & Westinghouse Brake & Signal Co. Ltd. *Brit. Pat.* 759,961

Vacuum Gasket at Low Temperatures

136/II

United States. Soft aluminium wire has been used successfully as a gasket material for vacuum systems. It is of particular importance in low-temperature work and has proved to form a satisfactory seal in liquid helium I and II.

Note by A. H. Spees, C. A. Reynolds, A. Boxer & G. Pearson *Rev. Sci. Instrum.* **28**, Dec. 1957 1090

A Greaseless Vacuum Seal for Rotating Shafts

137/II

United Kingdom. This note describes a variation on the Wilson seal, in which the grease is dispensed with by using P.T.F.E. washers on a steel shaft, taking advantage of the very low friction between these materials. The model shown uses four washers of P.T.F.E., with highly polished steel spacers separating them, both washers and spacers being 0.062 inch thick. The shaft is smaller than the washers, giving a clearance of about 0.010 inch and the whole assembly is then compressed axially. The P.T.F.E. flows between the spacers to make a seal on the shaft. Seals have been used at temperatures up to 200°C and have shown no sign of leaks greater than 10^{-5} lusec.

Note by E. A. Billett & J. Bishop *J. Sci. Instrum.* **35**, Feb. 1958 70

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Abstract No. and References

138/II

Note by F. J. Loucks Jr. *Rev. Sci. Instrum.* **28**, June 1957 468-469

Unique Sliding Seal for a Vacuum Chamber

(Reference only)

139/II

Note by R. H. Hodder *J. Sci. Instrum.* **35**, May 1958 182

Vacuum or Low-Pressure Seal Utilising Modified Standard Refrigeration-Type Flare Tube Fittings

(Reference only)

140/II

Note by C. J. Meechan & A. Sosin *Rev. Sci. Instrum.* **29**, April 1958

Electrically-Insulating Thermally-Conducting Vacuum Seal for Low-Temperature Use

United States. It is often necessary to provide a good thermal path, in an electrically insulated medium, particularly in some low-temperature experiments concerned with irradiating materials at temperatures below 80°K. Often a thermal path is required through an evacuated vessel; glass-metal seals cannot be used due to the danger of cracks forming. Sapphire is used as the basic thermally conducting electrically insulating medium. To make a suitable seal, sapphire rods are cast in vacuum-melted copper or lead.

141/II

Note by V. A. Heathcote & W. E. Read *J. Sci. Instrum.* **34**, June 1957 247

A Demountable Seal for High Vacuum Work

United Kingdom. A demountable seal which can be baked at 450°C is made by using a proprietary make of copper steam jointing rings. The copper rings are compressed between two steel flanges; the shape of the cross section of a ring is such that it gives effectively two concentric, circular, knife-edge contacts with the steel flanges. A hole is then bored through one flange and a connecting tube fitted so that the space between the two knife-edges can be evacuated by a backing pump. A good turned surface on the steel flange was found to be adequate. Ni-plated steel and ' K ' Monel surfaces were also used successfully, but chromium plating was not satisfactory. Ring and flange dimensions are given for diameters up to 8 inch. As a result of the compression it appeared that the copper formed a diffusion braze with the steel or nickel.

142/II

Note by N. W. Robinson *J. Sci. Instrum.* **34**, March 1957 121

Bakeable High Vacuum Seals

United Kingdom. Plane flanges can be joined and baked repeatedly to 450°C using an all-metal seal which has been developed for use with high vacuum demountable apparatus. Two flat copper washers are separated by a steel ring which has annular knife edges formed on the upper and lower surfaces, and a peripheral groove to allow a cantilever action of the knife edges when under pressure. Suitable dimensions are given for a 4 inch ring so as to give the necessary stiffness in the steel to cause plastic deformation of the copper when the flanges are bolted together, and still exert a positive force on the copper during the annealing which occurs on baking and on subsequent cooling. The number of sealing surfaces can be halved by using one copper washer of U-section instead of the two flat washers. For joining small diameter pump lines the copper tube is spun over one flange to form its own washer and one knife edge only is used.

143/II

Article by H. A. Adam, S. Kaufman & B. S. Liley *J. Sci. Instrum.* **34**, March 1957 123-124

Indium Seals for Dismountable Vacuum Systems

United Kingdom. Metal seals are preferred to rubber and neoprene in demountable vacuum systems when hydro-carbon vapours must be avoided. A seal was required for conditions where no clamping is possible and compression of the seal is due solely to atmospheric pressure. Indium was chosen because of its softness and negligible vapour pressure at room temperature, and satisfactory seals were obtained with indium wire retained in a circular groove of trapeziform cross-section between wedge-shaped rings of aluminium alloy and rings of porcelain. Vacuum seals were also easily obtained with other combinations of surfaces *e.g.* steel-steel, steel-porcelain and steel-alloy. As the minimum load required to form a seal is 5.9 kg wt/cm. length of wire, the least diameter of tube using atmospheric compression alone appears to be 23 cm. Measurements of leak rates (which were found to be, at best, between 1.5×10^{-3} and 2×10^{-3} μlitres per sec.) for different surfaces of porcelain and glass show that a very clean surface of porcelain must be highly polished to effect a vacuum seal with an atmospheric load of 8.6 kg wt/cm., although a seal can be formed on the less regular surface of ground porcelian if it is not thoroughly clean, as grease or dirt seals the crevices in the surface. The method can be applied to the standard type of vacuum union using the indium gasket instead of a rubber washer, and screwing the two sealing faces together. There appears to be a relationship between the sealing mechanism and the hardness properties of indium and it is suggested that substantial bolting of sealing flanges used at high temperatures can be avoided by choosing a metal with the appropriate properties of hardness and creep at the operating temperature.

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Construction of Thin Mylar Gas Target Holders

144/II

United States. Mylar is used as a window for experiments in nuclear physics. In this paper a technique is described for making gas target holders having Mylar windows with a thickness of 0.00035 inch. A vacuum-tight cell may be readily produced.

Note by J. C. Corelli, M. Livingston & L. Seidlitz *Rev. Sci. Instrum.* **28**, June 1957 471-472

Simple High-Temperature Vacuum-Tight Mica Window

145/II

United States. The usual method of sealing mica windows for high vacuum work at temperatures over 100°C is to first seal the mica to a cup of specially treated metal using lead borosilicate glass as the sealing medium. The unit is later sealed to the system. In the new design, an annealed copper ring 0.015 inch thick whose external diameter is that of the mica is used as an O-ring. The copper ring is placed on the vacuum side of the mica and the two are clamped together between two stainless steel flanges by a symmetrical array of six clamping bolts so that the copper is pressed to a thickness of 0.010 inch. The copper then provides a vacuum-tight seal between the mica and the steel flange on which it rests. When using these windows in sealed-off tubes, mass spectrometer leak detector measurements failed to detect any leak. The chief advantages of this type of assembly are : (*i*) high bake-out temperature (700°C) when synthetic mica is used as compared with 550°C for the older type; (*ii*) no special equipment required in construction; (*iii*) the window may be removed and re-used after machining the flange on to which the copper is pressed; (*iv*) non-magnetic materials are used throughout.

Article by F. Sterzer *Rev. Sci. Instrum.* **28**, March 1957 208-209

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Mica Window Assembly for Use at Elevated Bake-Out Temperatures

146/II

United States. The use of mica for r.f. or observation windows in vacuum devices offers many advantages. However, conventional mica window seals have the disadvantage of being restricted to relatively low bake-out temperatures since they are usually sealed with a low-temperature solder glass. A mica seal which can be used at high bake-out temperatures is described.

Note by A. R. Strad *Rev. Sci. Instrum.* **29**, June 1958 533

29 — MISCELLANEOUS APPARATUS — 29

A Radioactive Ion sat on Gauge and Its Application to the Measurement of Latent Heat of Vaporisation

147/II

See Abstract No. : 48/II

Correlating Vapour Pressures and Other Physical Properties

148/II

United States. In this article a general equation and a nomogram are given for correlating vapour pressures, latent heats and other physical properties of substances. The equation is of the form

$$\log P = \frac{(L)}{(L^1)} \log P^1 + C$$

where P is the vapour pressure of the material, P^1 that of a reference substance (water is the substance used in this article), L/L^1 the ratio of the molar latent heats of the material and the reference substance and C is a constant. The slope of the line, $m = L/L^1$, is independent of temperature and pressure. A table gives the values of log P^1 and L^1 for water over the temperature range 0°C to 350°C. A second table gives the values of m and C for over 500 organic compounds. The equation then enables P and L to be calculated at any desired temperature for which the data for water is given. A nomogram is also given which enables the same calculation to be performed. Instructions are given for using the nomogram for the above purpose and also, with the aid of further tables, for calculating Henry's law constant, the partial pressure of gases in liquids, partial pressures of adsorbed vapours are charcoal, etc. References are given to other papers which deal with further uses of the nomogram.

Article by D. F. Othmer, P. W. Maurer, C. J. Molinary & R. C. Kowalski *Industr. Engng. Chem.* **49**, Jan. 1957 125-137

The Measurement of Small Vapour Pressures

149/II

Italy. The author describes (and gives illustrations of) several instruments typical of the static and dynamic methods used in the determination of vapour pressures. Besides U-tube manometers and the Knudsen manometer, the Rodebusch manometer is described, where the vapour presses a non-return valve against its seating, on the other side of which a high vacuum is maintained. The valve is then opened by an electromagnet and the current necessary to open the valve is a measure of the vapour pressure. The author then gives the results of measurements undertaken with the McLeod on various substances, *e.g.* benzole, toluol, etc., and at the end of the article gives a table where the various instruments and measuring methods are listed with a short comment on their characteristics.

Article by G. Milazzo *Chem. Ing. Tech.* **28**, Oct. 1956 646-653

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150/II

Article by M. W. Cook *Rev. Sci. Instrum.* **29**, May 1958 399-400

Vapour Pressure Apparatus

United States. A vapour apparatus has been developed which has certain manipulative and accuracy advantages over the well-known isoteniscope. A reliable criterion for complete degassing is inherent in the apparatus. Since degassing is required only once for a series of determinations at various temperatures, the apparatus is well adapted where the amount of sample is limited. The useful temperature range of this apparatus is between the freezing point of mercury (–38.9°C) and the temperature at which the vapour pressure of mercury becomes appreciable (~0.1 mm. at 80°C). The apparatus may not be used if the sample reacts chemically with mercury.

(*Author*)

151/II

Letter by A. H. Price *Nature* **181**, 25.1.1958 262

Vapour Pressure of Tritiated Water

United Kingdom. Reports the determination, apparently for the first time, of the vapour pressure of HTO in the range 25–80°C. Solutions of various activities were prepared and allowed to come to equilibrium with their vapours in a glass apparatus (not described) which was totally enclosed in a thermostat. The vapour was then isolated from the liquid in each case and analysed by the butane method of Glascock, the ratio of activity in the vapour to that in the liquid being found to be constant at any one temperature, in accordance with Henry's law. This ratio can be shown to be equal to the ratio of the s.v.p. of HTO to that of H_2O. The results show this ratio to vary from 0.77 at 26°C to 0.89 at 76°C. A graph of $\log_{10}$ (s.v.p.) against 1/T is extrapolated to show tritiated water to have a lower b.p. than ordinary water. It also has a higher molar heat of vaporisation (10.9 against 10.3 kcal).

152/II

Vapour Pressure of Plastic Materials

See Abstract No.: 107/II

153/II

Article by J. Conard & J. Cabannes *C.R. Acad. Sci. Paris* **244**, 2.1.1957 52-54

Safety Assembly for Permanent Vacuum Installations Connected to Pumps

France. The article describes a safety device enabling the electronic control of a Penning vacuum gauge, the circulation of the cooling water and also to guard against a failure of the mains. Details of the design are given.

154/II

C. R. E. Legg *Brit. Pat.* 766,361

Improvements in Vacuum Control Systems

United Kingdom. The object of the device is to provide an electrically operated control for a vacuum system so as to safeguard the system in case of mains failure and which will always operate the backing line leak valve and the main valve to the chamber in the correct sequence and with suitable time intervals. The requirements may be summarised as follows : Before starting the pump the main valve to the chamber must be in the closed position whilst the backing line leak valve must be open. On starting the motor the leak valve must close immediately so that the backing line shall be roughed out before the chamber valve is opened. This routine is ensured by introducing a suitable time delay before energising the d.c. solenoid operating the chamber valve. A resistance capacity network in the rectified supply to the solenoid delays the closing of a single contact in this line for an interval during which the backing line is evacuated. On switching off or in the case of mains failure the pump stops and the d.c. supply to the chamber valve solenoid is cut off immediately. The chamber valve closes and safeguards the vacuum system. The leak valve however does not open immediately since it is held in the closed position whilst a condenser across its d.c. operating solenoid discharges through the solenoid winding. The leak valve then opens leaking air into the backing line and preventing oil being drawn up from the backing pump.

155/II

Article by C. J. Penther *Rev. Sci. Instrum.* **28**, June 1957 460-463

Vactroller—A Laboratory Vacuum Controller

United States. Pirani gauges are used in an a.c. bridge circuit to sense deviation from the set point and with the aid of a proportional leak valve, control the pressure of laboratory-scale vacuum systems over the range of 0.1μ to 10,000μ Hg. Pressure fluctuations from the controller action are negligible and stability of +1% has been obtained over long periods without operator attention. Set point is easily adjustable by panel control knob.

(*Author*)

156/II

Schlumberger Well Surveying Corp. *Brit. Pat.* 779,913

Apparatus for Controlling Gas Pressures

United States. A new method is described for maintaining a constant pressure of hydrogen, or its isotopes, in a partially evacuated chamber. It avoids the bulky apparatus of conventional methods which use an external gas supply and evacuating system. The method depends on the fact that suitably treated zirconium filaments can be caused to absorb or emit gas according to the degree to which they are heated. A typical gas control apparatus as applied to an ion source is described.

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Abstract No. and References

Leak Detector for Hermetic Seals

157/II

Canada. A device for rapid non-destructive testing of hermetically sealed components to a sensitivity comparable to that of a mass spectrometer is described. The components are placed in pouches formed by drilling holes in a thick steel plate of 8½ inches diameter. Another rectangular plate is ground to fit on top of the thick plate having one hole of the same size as the pouch holes such that rotation of the lower plate brings the pouches successively in position below it. The top plate is fixed and has a number of grooves cut in it which connect the pouches with different vacuum pumps as the lower plate is rotated. Also the top plate has other channels to provide a guard vacuum which is kept at 0.7 mm. Hg by an auxiliary pump. The contacting surfaces of the two plates are lubricated with Apiezon-C oil. Components are first cleaned in trichlorethylene and warmed on a hot plate. One is then placed in the pouch accessible through the hole in the top plate and the lower plate is rotated. As each pouch appears below the filling hole a component is placed in it. The sequence of events on rotating the plate is such that each pouch is evacuated first by the auxiliary pump, then by the fore-pump of two small diffusion pumps then by the first of the small diffusion pumps for three time intervals of indexing by which time the pressure is reduced to the order of 1×10^{-5} mm. Hg. In the next position of the pouch it is evacuated by the second of the diffusion pumps to a pressure of $2\text{–}5 \times 10^{-6}$ mm. Hg. After 10–15 seconds a valve is closed cutting off the pouch from the pumps and the pressure rise is observed on an ionisation gauge connected *via* a cold trap to the pouch. If the rise of pressure is in excess of that measured due to normal outgassing the components is rejected as leaking. Large leaks can be detected either by the fast rate of rise on closing the valve or by the fact that the pressure in this position will not reach $2\text{–}5 \times 10^{-6}$ mm. Hg after 15 seconds pumping.

Article by B. W. Schumacher *Electronics* **30**, March 1957 284-294

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Leak Testing of Vacuum Plant by Helium Analysis

158/II

Gt. Britain. This article describes an apparatus for locating and measuring leaks in a plant handling gas at reduced pressure. The principle of the method is to remove a sample of the gas from the plant and to analyse it for the presence of helium and neon, absent from the plant gas, but carried in by any leakage from the air. It is claimed to be simpler, cheaper and more stable than methods using mass spectrometers. A standard sample of gas is taken from the plant and passed through an adsorption column containing charcoal cooled to liquid N_2 temperature. When the pressure beyond this column has built up to a pre-determined level, a manometric switch operates a set of electromagnetic valves and the gas which has passed through the first adsorption column is allowed to enter a second, shorter, column. Finally, the light gas fraction emerging passes through a further small charcoal column, cooled in liquid N_2, which acts as a getter, and passes into an ionisation gauge. The gauge, previously calibrated against standard samples, measures the pressure of the light fraction containing the He and Ne from the in-leaking air. To use the equipment, it is first evacuated and the charcoal columns baked at 300°C. The gas sample is then admitted to the reservoir and brought to standard pressure by the addition of pure N_2 or O_2. The sample is controlled in its passage through the apparatus by electromagnetic valves to a standard schedule and the sudden increase in ion current when it reaches the gauge is measured by a galvanometer. The limiting sensitivity corresponds to about 0.01 ml. of air at N.T.P. in a sample of 1.31 of plant gas.

Article by E. Glueckauf & G. P. Kitt *J. Sci. Instrum.* **35**, June 1958 220-223

A Simple Vacuum Leak Detector Using a Radio-Frequency Mass Spectrometer

159/II

Hungary. In the radio-frequency mass spectrometer an ion beam of high velocity passes through a series of plane-parallel grids. Alternate grids are grounded and the others have a radio-frequency voltage applied to them. If the time of transit of an ion between two grids is approximately half of the period of the r.f. oscillation, this particular ion gains energy from the r.f. field. All the ions then encounter a retarding potential barrier which allows only ions of the highest energy to pass through to the collector. By suitable choice of voltages it can be arranged that only ions of a particular gas will contribute to the collector current. A leak detector making use of this principle is described; it is much simpler and less expensive than the magnetic mass spetrometer leak detectors while still being capable of detecting leaks of the order of 2.8×10^{-8} l mm./sec. over the pressure range 10^{-3} to 10^{-7} mm. It is also possible to use the instrument as a conventional hot-cathode ionisation gauge. The associated electronic equipment consisting of an electron emission stabiliser, a d.c. amplifier for the collector current and an r.f. oscillator, (operating at 7 mc/s. if the probe gas is hydrogen) is portable. The glass gauge head is 70 mm. long by 35 mm. diameter and is suitable for sealing to any kind of vacuum system.

Article by P. F. Varadi & L. G. Sebestyen *J. Sci. Instrum.* **33**, Oct. 1956 392-393

Spectral Analysis-Type Leak Detector

160/II

Japan. Preliminary work on a spectral analysis-type leak detector is described. The detector consists of a discharge tube, a spectrometer, a photomultiplier and an amplifier. An electrodeless discharge tube which is attached to the vacuum system is operated by a high frequency current of about 100 megacycles modulated by 60 cycles. The emitted light from the discharge tube is dispersed by a constant deviation prism spectrometer and received by a photomultiplier IP 21. The audio frequency parts of the signals from the IP 21 are amplified and rectified so as to operate a d.c. milliammeter. When small quantities of air, hydrogen or helium are introduced to the vacuum system through an adjustable leak valve, several peaks corresponding to the spectral lines characteristic of the gases appear on the meter. In the present work, the leak rates detectable in practice are about 10^{-4} litre mm./sec. for hydrogen and 10^{-5} litre mm./sec. for helium. Compared with the mass spectrometer-type leak detector the new detector has the following advantages : (*1*) No need for a vacuum system in the detector, (*2*) portable and durable construction, (*3*) simple circuit and rapid operation.

(*Authors*)

Article by M. Sakisaka, Y. Oyama & T. Furushima *J. Japanese Vac. Soc.* **7**, April 1957 121-130

Abstract No. and References

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161/II

An Evaporated Gold Bolometer

United Kingdom. Describes the construction and performance of a bolometer intended as an infra-red detector for wavelengths beyond the sensitivity region of photoconductive cells. It consists of a very thin strip of blackened gold, 2.6 mm. × 0.2 mm. × about 0.03 micron, supported by a thin film of cellulose acetate stretched over a slot in a strip of mica. The resistance is about 15 ohm and the temperature coefficient about 0.2% per degree C. Attempts to produce a bolometer by evaporating tellurium were abandoned because the elements were noisy; tin, copper and silver were also unsatisfactory. Preparation of the cellulose acetate film and its mounting on the mica strip are described. Gold electrodes are first evaporated on to the ends of the mica strip and then a very narrow strip of gold is evaporated over the electrodes and the cellulose film. It is blackened by a further evaporation of gold, carried out in N_2 at a pressure of 1.75–2.0 mm. Hg. There is a sketch showing the mask used for evaporation of the element, but no description of the vacuum system. For testing, the bolometer was mounted in a brass container with a rocksalt window, which could be evacuated. Fine wire leads were soldered to the gold electrodes with indium. The resistance was measured by a Wheatstone bridge circuit and found to decrease about 10% with ageing, to about 15 ohm. To measure the sensitivity, the bolometer was allowed to receive radiation from a ' black body ' (a blackened, solid brass cylinder) at 450°K, *via* a rotating sector disc to ' chop ' the energy at 10c./sec. The output was coupled to a high-gain, narrow-band amplifier by a matching transformer. The sensitivity in air is given as 0.43 V/W, in vacuum as 1.5 V/W and the minimum detectable energy (for unity signal/noise ratio) as 3.6×10^{-10} W, measured at 10 c./sec. with 1 c./sec. band width. The time constant was found to be 5 m.sec. in air and 7 m.sec. in vacuum.

Article by E. Archbold *J. Sci. Instrum.* 34, June 1957 240-242

162/II

Improvements in Ionisation Chambers

France. The invention relates to an ionisation chamber in which the problem of electrical leakage between the electrodes is reduced by incorporating the source of voltage between the electrodes within the ionisation chamber. One electrode constitutes the outer casing of the chamber which may be metal or some non-conducting material with an internal metal coating and the other electrode is concentrically placed within the first and contains a suitable battery, for example a Zamboni pile, the positive terminal of which is connected to this electrode and the negative terminal taken through an insulator of polystyrene situated in the end wall of the chamber to the exterior of the chamber. A resistance is connected either inside or outside the chamber between the negative voltage line and earth to which is also connected the outer electrode or case of the chamber. The chamber contains a suitable gas at a low pressure in which the incidence of any radiation causes ionisation within the chamber which in turn causes a current to flow in the resistance, this developing a voltage across it, which voltage can be caused to influence some external circuit or apparatus. In the structural embodiment described contact between the end wall of the chamber, which is earthed, and the interior surface of the chamber is ensured by means of spring contacts fixed firmly to the end wall and pressing onto the interior surface of the chamber contacting the metallic coating. The central electrode containing the Zamboni pile is supported by the Zamboni pile, the negative terminal of which is supported rigidly through the insulator in the end wall which support also serves as the negative terminal of the battery and against which on the external side a spring presses which is electrically connected to a pre-amplifier which is built onto the end of the chamber. The positive side of the battery is connected rigidly to the inner electrode which it supports. The voltage of the battery is, for example, 200 V. By this construction no voltage appears across the insulator until a signal appears and then it is only a very small voltage so that the material of the insulator is not subjected to any substantial voltage and consequently will not transmit any electrical leakage.

C.E.A. *Brit. Pat.* 780,283

163/II

Luminescent Gas Flow Visualisation for Low-Density Wind Tunnels

United States. Conventional flow visualisation techniques depending on charges in refractive index of gas are of no use when pressures concerned are of the order of 0.1 mm. mercury. In their place the authors describe a new technique which uses observation of the visual light which accompanies certain gaseous reactions. In the case of nitrogen this effect is thought to be due to atomic nitrogen which may be present to one per cent in the molecular gas. A more useful example of this which can be described as chemi-luminescence takes place in air or any mixture of nitrogen containing a few per cent oxygen in which the reaction consists of the oxidation of nitric oxide by atomic oxygen to nitrogen peroxide with the emission of visible light. Of the noble gases a brilliant after-glow has been observed with argon and a less brilliant one with helium. The authors feel that in these cases the after-glow is due to some dissociative recombination of molecular ions. In their use in Low Pressure Wind Tunnel work all the gases are, prior to being ejected through the nozzle, passed through a region in which an electrodeless discharge is maintained and in the case of air and nitrogen a subsidiary bleed of nitric oxide is available near to the ejection nozzle. For wind tunnel speeds of up to nearly Mark 4 this system has proved successful but above this, as the authors show by mathematical treatment and experiment, it has proved unsuccessful due principally to the large increase in stagnation pressure with its resultant effects on collison frequency and mass flow. The intensity of the after glow depends on concentration, collision frequency and reaction probability, per collision of the particles involved. Of these the most important is collision frequency which ultimately varies with gas density and temperature. The equipment used by the authors in the University of California wind tunnel is described. The excitation discharge is produced in a side arm 15 cm. in diameter and 90 cm. long upstream from the test section and near to the nozzle. The oscillator was of the Colpitts type with a large aluminium strap for coupling into the discharge space. Several photographs are included showing after glow gas flow visualisation effects at speeds from Mach 1.7 up to Mach 3.8 in mixtures of gas including argon, helium, nitrogen and air. For the higher speeds air was used as the useful range of argon and helium was limited to Mach 2 and that of nitrogen to Mach 3. The authors summarise the paper and conclude that the technique described could have applications to higher Mach numbers with better tunnel geometry and improved excitation technique.

Article by W. B. Kunkel & F. C. Hurlbut *J. Appl. Phys.* 28, August 1957 827-835

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Abstract No. and References

Cryostat for Reactor Irradiation

164/II

United States. A cryostat for continuously bathing samples in liquid nitrogen or other heat transfer liquids during nuclear reactor irradiation has been constructed and successfully operated. The samples to be irradiated at low temperature are immersed in the heat transfer fluid which is high-purity liquified nitrogen circulating in a closed system at a pressure greater than that of the atmosphere. The liquid is kept below its boiling point throughout its cycle by use of a heat exchanger located outside the reactor shield. The heat exchanger is cooled by commercial liquid nitrogen boiling at atmospheric pressure. Since oxygen and water vapour from the air cannot enter the closed pressurised system, no chemical explosions inside the nuclear reactor have occurred. Baths at other temperatures may be obtained by substituting suitable liquids for the high-purity nitrogen heat transfer fluid and by keeping the heat exchanger at the desired temperature.

(*Authors*)

Article by C. C. Sartain & H. P. Yockey *Rev. Sci. Instrum.* **29**, Feb. 1958 118-121

Handling Compressible Fluids in Chemical Processing

165/II

United States. Compression equipment and suction pumps of various types are widely employed in handling compressible fluids in the chemical industry. Some typical applications include : Regulation of the pressure in chemical reaction vessels above or below atmospheric, movement of gases for purposes of liquefaction or for storage in cylinders and tank cars, provision of compressed air for tools and machinery, and compression of various fluids in refrigerating plant. A chart shows the range of capacities covered by four main types of compressor, which are in ascending order of capacity (*a*) reciprocating, (*b*) rotary displacement, (*c*) centrifugal, and (*d*) axial-flow compressors, the last-named having inlet capacities up to about 3×10^6 c.f.m. Another table gives approximate figures for the maximum discharge pressure of the above four types, reaching about 35,000 p.s.i. for a reciprocating compressor, and the section pressures which may be expected from reciprocating and rotary vacuum pumps, steam jet ejectors, and oil diffusion pumps. Also tabulated are the maximum pressures commonly found in a number of industrial processes, ranging from about 15 p.s.i.g. for refrigeration compressors using Freon-11 to about 30,000 p.s.i. in polyethylene synthesis. A number of modern chemical processes are then described in which compressors or pumps play an important part, and any special problems encountered are discussed. For example, oil-lubricated compressors may not be used in the compression of oxygen for liquefaction, or in processes where catalysts may be poisoned by lubricating oil. In these cases reciprocating compressors fitted with carbon packing rings may be satisfactorily employed. Schematic flow diagrams of some of the processes mentioned, and photographs of typical compressor installations, are also shown. Modern trends in compression methods are noted, together with brief guides to economic selection of equipment, an important point in processes such as the synthesis of ammonia, where compressor costs may amount to as much as 20% of the total plant cost.

Article by P. R. Des Jardins *Chem. Engng.* **63**, June 1956 178-187

Rotary Compressors and Vacuum Pumps

166/II

United States. This short review article describes and compares the characteristics of the three main types of positive-displacement rotary compressors used in industry. These are (1) sliding vane types, in which longitudinal vanes slide radially in a rotor mounted eccentrically in a cylinder, (2) rotary lobe types, where two mating lobed impellers, driven by external gears, revolve within a cylinder, and (3) liquid-ring types, in which a circular impeller with fixed blades rotates inside an elliptic casing partly filled with water or other liquid. Figures are quoted showing the sizes of compressors available commercially and the pressures reached by single or multi-stage versions. Rotary lobe compressors, for instance, may have capacities up to 50,000 c.f.m., and sliding vane types may produce pressures up to 400 p.s.i. Vacuum performance figures are also given, the limit in most cases being about 29 inch Hg. Since the three types dealt with are all of positive displacement, methods of controlling the discharge pressure are discussed, such as relief valves or by-pass controls. Other points mentioned briefly are the problems of lubrication and handling of corrosive fluids.

Article by W. L. Bruce & W. A. Schubert *Chem. Engng.* **63**, June 1956 224-226

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(Reference only)

1/III
Letter by R. T. Spurr & J. G. Butlin
Nature
179, 8.6.1957
1187

Ionic Bombardment Cleaning of Glass

2/III

United Kingdom. The use of the glow discharge for cleaning glass surfaces by positive ion bombardment is discussed. Experiments are described in which glass surfaces are bombarded with positive ions or electrons. It is concluded that electrons formed in a glow discharge can decompose hydrocarbon molecules absorbed on surfaces which they strike, whereas positive ion bombardment removes surface contaminants from glass, and at high current densities may remove components from the glass itself.

Article by L. Holland
Nature
181, 24.5.1958
1451-1452

Calculator for Thin Film Measurements

3/III

See Abstract No.: 154/I

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Note on the Limits of the Polarimetric Method of Thin Film Measurements

4/III

Czechoslovakia. This note seeks to refute a criticism made by R. D. Mattuck (*J. Opt. Soc. Amer.*, **46**, 1956, 615) of a statement made by the author (*J. Opt. Soc. Amer.*, **37**, 1947, 929) to the effect that the polarimetric method is able to indicate the existence of thin films having atomic dimensions and should permit accurate measurements for a film of thickness 10 Å. The note discusses the accuracy of the method for films of various refractive indices and various thicknesses. In particular, in the most usual case where the refractive index of the film is less than that of the glass substrate it is possible to evaluate thickness from 100 Å to a few Angstroms, provided the refractive index of the film is known approximately.

Letter by A. Vasicek
J. Opt. Soc. Amer.
47, June 1957
565-566

Two-Colour Monitoring Method for Reproducing Thin-Film Thicknesses

5/III

See Abstract No.: 156/I

Nondestructive Interferometric Thickness Measurement of Thin Transparent Films

6/III

See Reference No.: 151/I

Thickness Measurement of Epitaxially Grown Films

7/III

United Kingdom. Rock-salt is frequently used as a crystalline base for the deposition of a thin film for epitaxial study, since it is easy to remove the film by dissolving the base away. Measurement of the film thickness is difficult because a cleaved rock-salt surface is rarely flat enough for using the method of multiple-beam interferometry. In the method described here, the film is floated off the rock-salt and picked up on an optical flat, the edge of the film is silvered and multiple-beam interferometry is carried out in the usual way.

Letter by O. S. Heavens & D. C. Pandeya
Nature
181, 17.5.1958
1394

The Multiple Scattering of Positrons and Electrons

8/III

United Kingdom. Differences have occurred between the theoretical and experimental results obtained for the multiple scattering of positrons and electrons by gases or thin films at energies below about 1 MeV. Experiments appeared to show that the scattering was less than that predicted by the theory of Moliere, and that the differences between the distribution width for electrons and positrons were greater than the values to be expected from the calculations of Mohr. A large β-ray spectrometer has been used to provide further information on these topics, measurements being made with particles having energies of 4 MeV. The spectrometer provides a strong flux of either positrons or electrons in the form of a convergent hollow conical beam, the particle source being a thin pure copper disc which had been activated for one week by neutrons in the Harwell atomic pile. Photographic emulsions and thin metallic films were used as scattering targets. The films were prepared by vacuum evaporation of pure gold or silver from an eight-filament jig onto a thin Melinex film. The eight sources gave a highly uniform deposit over the region to be used in the experiment. The particle detector used was an anthracene crystal mounted on a long Perspex light pipe which was terminated by a photomultiplier located outside the magnetic field of the spectrometer. The detector could move along the axis of the scattered beam, within the outer vacuum tube of the spectrometer. A careful procedure involving frequent stability checks and background counts was followed in such a way that, in the course of

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Article by C. Henderson & A. Scott *Proc. Phys. Soc.* 70, March 1957 188-194

the electron-positron ratio determination, the uncertainties in the decay exponent of the source were minimised. A Perspex charged-particle absorber was used to determine the amount of background gamma radiation. The experimental and theoretical Moliere distributions for counts along the axis were found to be in close agreement. The values obtained for the electron-positron ratios were, in general, compatible with extrapolations of the calculations of Mohr and of Tassie. The differences for the two types of particle were much smaller than those reported by Groetzinger, Humphrey and Ribe for scattering in argon.

9/III Article by L. M. Rieser Jr. *J. Opt. Soc. Amer.* 47, Nov. 1957 987-994

Reflection of X-Rays from Condensed Metal Films

United States. The results of a series of measurements on the x-ray reflectivity of metal films evaporated on a glass substrate are presented. Comparison of these results with theory is made and the necessity of making measurements on bulk material is pointed out if the disagreements with theory are to be eliminated.

(*Author*)

10/III

Formation of Luminescent Films by Evaporation

United States. During the evaporation of already activated phosphor materials in vacuum, the activator is not always lost but probably exists as a separate phase in the deposited film. It is shown that by baking these films deposited on quartz or glass, good crystalline formation is induced in an otherwise partially amorphous film, and that the activator impurity diffuses into the crystal structure. Most success has been achieved with manganese activators in zinc sulphide, calcium fluoride, zinc phosphate and zinc silicate. Limited success has been obtained with silver and copper-activated phosphors. It is considered that the good results with manganese are due to the relatively large quantities required to give activation. Such a material is less affected by impurities. The substrate material is important in producing good quality films, and for best results the glass must have a high softening point and coefficient of expansion similar to that of the phosphor. The brightness of a transparent phosphor film is limited by internal reflection, and for high refractive index materials such as zinc sulphide, as much as 95% of the total light may be lost. However by deliberately making fogged or grainy films, light outputs as high as those of the original phosphor powder have been obtained, with much finer resolution. A demountable cathode ray tube was made for testing the films and the brightness was measured from the gun side using a 'Spectra Brightness Spotmeter'. Measurements of spectral emission were also made. The films were deposited by evaporating given quantities of powder to completeness. The pressure during evaporation was of the order 5×10^{-5}mm. Hg and a liquid nitrogen trap was used. The substrates were outgassed in vacuum by heating prior to evaporation. Of the films studied, manganese-activated zinc silicate showed the most promise with regard to brightness and transparency. Tab. II showing the evaporation and processing techniques is reproduced here.

Article by C. Feldman & M. O'Hara *J. Opt. Soc. Amer.* April 1957 300-305

TABLE II

Phosphor Material	*Filament*	*Filament Temperature °C*	*Evaporation Time Mins.*	*Substrate*	*Post-firing conditions*		
					Atmosphere	*Temperature*	*Time Mins.*
2nS (Mn)	Ta boat	1,050	60	N.B.S. special glass F.83	Vacuum(He)	750–800	5–15
CaF2 (Mn)	Ta boat	1,550	5	Quartz glass	Vacuum	600–700	5
ZnF_2 $(PO_4)3$	W basket	1,525	20	Quartz	Air(O_2)	850	10
Ca WO_4 (W)	W wire	2,000	3	Pittsburgh noncorrosive microslides	Air(O_2)	550	120
Zn_2 Si O_4(Mn)	Ta boat	1,300	30	Quartz Vycor	Air	1100	180

11/III

Optical Properties and Oxidation of Evaporated Titanium Films

United States. The preparation of films of pure titanium, by evaporation under vacuum better than a 5×10^{-6}mm. Hg is described and the optical properties and oxidation of such films is discussed. Fast evaporation of titanium under very high vacuum is necessary to produce pure compact films. The gettering action of the metal was made use of in producing the vacuum required by using two evaporation sources, one, consisting of titanium, for lowering the pressure and the other for deposition. Films were deposited at up to 100 Å per sec. at a distance of 20 cm. Using the titanium gettering source, films of Ag, Au and other metals could be prepared at these low pressures as low as $1–2 \times 10^{-6}$mm. Hg. Optical constants were determined by Drude's polarimetric method, and calculated reflectance values agreed well with those observed. Films of titanium of a thickness of 100–300 Å were found to have quite uniform transmittance throughout the visible spectrum. At room temperature the rate of oxidation of titanium equals that for aluminium, *i.e.* about 15 Å of oxide film after two hours exposure to air, reaching a maximum of about 35 Å after one month. Oxidation of titanium at 300°C begins at eight times the rate for aluminium.

Article by G. Hass & A. P. Bradford *J. Opt. Soc. Amer.* 47, February 1957 125-129

12/III

Titanium Dioxide Beam Splitters

United Kingdom. Titanium dioxide beam splitters of large surface area have been made by heat oxidation in air of evaporated titanium films on glass. It has been observed that a reaction between the glass and the titanium may take place during the heat-oxidation, causing the oxide film to craze. By means of careful experimental work, it was established that the crazing only occurred at temperatures above about 400°C,

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and was in no way connected with the cleaning or evaporation conditions, full details of which are given. The reaction was unpredictable so that there was always a risk of the film crazing when making beam splitters of optical thickness $\lambda/4$, which required temperatures of 400°C to produce complete oxidation. It was considered that the effect arose from titanium reacting with certain constituents in the glass surface which were present to various degrees. This was confirmed by the use of barrier layers between the glass and the titanium. Aluminium oxide films in the thickness range 650 to 1,400 Å prevented the reaction at 400°C. Below 350 Å the protective effect of the aluminium was lost, and a bad reaction could occur. However, aluminium oxide is difficult to evaporate and control, and experiments were continued with silica barrier layers, prepared by the slow evaporation of silicon monoxide with satisfactory results. Magnesium fluoride films were also used with success, but the combination of Mg F_2/TiO_2 after heat oxidation was softer than the single-layer TiO_2 film, but would be sufficiently durable for most optical uses.

Letter by L. Holland T. Putner & S. Bateman *J. Opt. Soc. Amer.* 47, July 1957 668-669

Method of Producing Titanium Dioxide Coatings

13/III

United States. This invention relates to the commercial production of a durable, transparent coating of titanium dioxide on the surface of optical elements by evaporating titanium in a near vacuum of oxygen. An ordinary bell jar arrangement is used. The process has the advantages that the coatings do not require baking to convert them to the clear dioxide and their thickness can be measured according to the colour of the light reflected from them. Their index of refraction is about 2.4. The optical element may be a lens or a flat glass surface or a surface which already carries one or more coated layers of high or low index materials, for example an optical interference filter. Metallic titanium is preferred as the vapour source and is best supported in a boat consisting of a simple dimple in a ribbon of tungsten. Titanium oxides can be used, however, particularly those which evaporate easily. An atmosphere of substantially pure oxygen is required and the pressure must be chosen so that it is high enough to ensure oxidation of the titanium molecules but not high enough to oxidise the titanium source or to interfere with the rate of coating. Pressures between 2×10^{-4} and 3×10^{-3}mm. Hg have been found suitable. The optimum pressure within this range depends on the distance between the evaporating source and the optical elements. A consideration of the mean free path of titanium molecules in oxygen indicates that the most suitable range of pressure is that in which the mean free path of molecules in the oxygen atmosphere is between 0.2 and 1.0 times the distance of the optical element from the source. In practice the pressure is maintained below 3×10^{-3}mm. Hg but above the pressure at which the mean free path of molecules in the oxygen is equal to the distance of the optical element from the titanium source. The bell jar is evacuated in the usual manner throughout the evaporation process and oxygen is continuously admitted to compensate for that lost by combination with titanium and by pumping. For routine production it has been found best to admit oxygen near the base plate so that it is uniformly diffused by the time it reaches the optical surfaces. These surfaces can be equidistant from the source on a concave support. With this arrangement, and with the titanium source centrally located at the bottom of the bell jar, the presence of the oxygen gives rise to a slightly thicker coating on the more centrally-situated optical elements. The differences in thickness are, however, well within manufacturing tolerances. In some cases faster coating has been obtained by introducing oxygen through a pipe at a higher level in the bell jar. Special arrangements of orifices and screens can also be used to provide uniform coatings. If the optical elements are supported so that they are not equidistant from the source, *e.g.* in a flat plane, suitable masks are required to ensure uniform coatings. One application of the process is for coating an optical element with alternate layers of titanium dioxide and a low index compound, for example magnesium fluoride. The low index compound is evaporated in a near vacuum of less than 10^{-4}mm. Hg. Substantially pure oxygen is then introduced to maintain the pressure between 2×10^{-4} and 1×10^{-3}mm. Hg while evaporating metallic titanium. The process is repeated to give the required number of coatings. If necessary the elements can be moved to separate bell jars for coating the alternate layers.

D. S. Brinsmaid W. J. Keenan G. J. Koch W. F. Parsons & Eastman Kodak Co. *U.S. Pat.* 2,784,115

Optical Properties and Structure of Cerium Dioxide Films

14/III

United States. In the production of multilayer all-dielectric optical interference filters, there is a need for a stable material of high refractive index. Possibilities include TiO_2 and CeO_2. Unfortunately TiO_2 cannot be easily evaporated to produce a non-absorbing film, but CeO_2 can be deposited to give a durable non-absorbing film of high-index. This paper gives data on the optical properties and structure of CeO_2 films prepared under various conditions. Their usefulness in multilayer film combinations is demonstrated. The experimental techniques are described in detail. Tungsten evaporation sources were used, which were shuttered during the pre-heat period to avoid contamination of the work. The temperature of the glass or fused quartz substrates could be raised to 350°C by means of a radiant heater, and the pressure in the vacuum chamber could be maintained at 10^{-5}mm. Hg. The evaporation could be controlled by measuring transmittance or reflectance of monochromatic light during deposition. The absolute thickness measurements were made using multiple-beam interferometry. Accurate optical measurements of reflectance and transmittance were also made from 220 μ to 1,000 μ using modified Beckman Models DU and DK Spectrophotometers. The structure of CeO_2 films was examined by electron diffraction. Films produced by direct evaporation of CeO_2 were found to be durable and non-absorbing in the visible, making them useful for beam splitters or high-index films in multilayer film combinations. The refractive index of CeO_2 films was found to depend upon the temperature of the substrate. An increase of the substrate temperature from 25°C to 300°C increases the refractive index of the condensed film from about 2.2 to 2.4 at $\lambda = 550$ μ. The absorption of CeO_2 films increases greatly below 400 μ and reaches a maximum at 300 μ. Evaporated CeO_2 films show the same cubic structure as the bulk material and crystallise in no preferred orientation on amorphous substrates. The crystal size in the condensed films increases with increasing temperature.

Article by G. Hass J. B. Ramsey & R. Thun *J. Opt. Soc. Amer.* 48, May 1958 324-327

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15/III

Improvements i.a.r.t. the Production of Thin Layers

United States. Sputtered films of CdO on transparent supports such as glass, mica or Perspex for the production of electrically heated windows, observation ports, etc. suffer from the disadvantages that the electrical resistivity of the film varies with time and/or temperature due to chemical action with the atmosphere and also that some light is lost in transmission by reflection at the interfaces. These defects are overcome by evaporating an additional transparent layer on to either face of the film. The deposition of As_2O_3, MgF_2 or cryolite is preferred to produce a 'bloomed' surface at the substrate-film interface, while SiO is preferred for the film-air interface. The production of both types of additional layer is also claimed. The specification also contains a description of the apparatus used which incorporated a drive motor to move the substrate to and from the evaporating source, and photo-electric measuring means for determining the thickness of the deposited films.

G. A. Veszi & P. Markus
Brit. Pat. 771,231

16/III

Some Properties of Thin Metallic Films II. Study of Thin Metallic Gallium Films

See Abstract No.: 160/I

17/III

Reflectance of Evaporated Aluminium in the Vacuum Ultra-Violet

See Abstract No.: 162/I

18/III

Influence of Purity, Substrate Temperature and Ageing Conditions on the Extreme Ultra-Violet Reflectance of Evaporated Aluminium

See Abstract No.: 161/I

19/III

The Spectral Reflectivity of Back-Surface and Front-Surface Aluminised Mirrors

United Kingdom. Results are given of a determination of spectral reflectivity of back and front aluminised quartz mirrors in the wavelength range 0.25–1.35 μ. A résumé is given, including reference to other recent work on front-surface mirrors, showing what are thought to be the highest values of reflectivity obtainable over the range 0.1–12 μ. The technical precautions necessary for the attainment of these optimum valves are discussed in practical terms.

(*Author*)

Article by G. G. Twidle
Brit. J. Appl. Phys.
8 August 1957
337-339

20/III

Infra-Red Reflectance of Evaporated Metal Films

United States. A high-precision method of measuring the reflectance of evaporated metal films is described and the results for aluminium and silver as a function of wavelengths (visible to 13 microns) and angle of incidence (20° to 60°) are reported. The apparatus was constructed as an accessory to the Perkin-Elmer Model 112 infra-red spectrometer using an NaCl prism. A well-collimated beam of infra-red radiation was reflected from two parallel glass plates of dimensions 3 inches by 10 inches which were coated with the surface to be measured. The intensity of the radiation after being reflected from the parallel mirrors, 2, 4, 6, 8, and 10 times was recorded by the pen and ink recorder of the spectograph and the reflectance was determined as a root of the ratios of these readings. The reflectances measured are accurate to 0.2%.

(*Authors*)

Article by D. M. Gates C. C. Shaw & D. Beaumont
J. Opt. Soc. Amer.
48, Feb. 1958
88-89

21/III

Infra-Red Absorption of Three-Layer Films

United States. The infra-red absorption of three-layer films as presented here is a generalisation of Hilsum's solution to the problem. This infra-red absorption is dependent on the optical parameters of the film, which in Hilsum's calculation were the same for the entrance and exit film. The present calculation considers different optical parameters for the entrance and exit film; an absorption of over 95% is obtainable.

(*Author*)

Article by P. A. Silberg
J. Opt. Soc. Amer.
47, July 1957
574-578

22/III

Anti-Reflection Coatings for Semi-Conductor Filters

United Kingdom. Several important infra-red filters use semi-conductors with refractive index near 4 (*e.g.* Ge, PbS, InSb) which have high reflection losses, so that blooming is necessary. The ideal blooming material would have a refractive index of 2.00 and $PbCl_2$, with an index of 1.9, is shown to be an improvement on previously used materials. Results show that blooming with $PbCl_2$ more than doubled the transmittance of an InSb filter in the range 10–13.5 micron. The method of grinding and polishing the semi-conductor crystal is briefly described.

Article by S. D. Smith & T. S. Moss,
J. Sci. Instrum.
35, March 1958
105

23/III

Interference Filters for the Infra-Red

See Abstract No.: 149/I

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Applications of the Interference of Light in Thin Films — 24/III

See Abstract No.: 147/I

Optical Properties of Magnesium Fluoride Films — 25/III

Japan. Magnesium fluoride films were prepared by vacuum evaporation from a molybdenum boat at pressures below 10^{-5} mm. Hg being protected from oil contamination by a CO_2 and alcohol cold trap. They were deposited on optical flats of crown glass and on fused quartz at a distance of 25 cm. from the boat. Evaporation was carried out at various substrate temperatures up to 350°C, temperatures being measured by a copper-constantan thermocouple embedded in the substrate. Thermal cleaning of the deposition surface was carried out by heating in vacuum for one hour at 350°C. Film thicknesses were measured by multiple-beam interferometry and transmittance and reflectance values were found using a monochromator and a photomultiplier tube and making use of a correction formula. The losses by optical scattering were thus compared. Films deposited on fused quartz at temperatures above 240°C showed appreciable scattering whilst those made simultaneously on glass at the same temperature showed no detectable scatter. The difference in behaviour is tentatively explained by the tendency of the fluoride to be mobile on the substrate surface during the initial stages of coating resulting in decreased adherence of the film to the substrate. Estimates of probable errors in measurement are made and give a possible error in scattering values of ± 0.008. The change of scatter with film thickness and variation of transmittance with wavelength are also examined. Ageing effects in the films were nearly always negligible with the exception of the films deposited on quartz at room temperatures. The causes of film porosity are briefly discussed.

Article by N. Morita *J. Phys. Soc. Japan* **11**, Sept. 1956 975-980

Optical Properties of Magnesium Fluoride Films in the Ultraviolet — 26/III

United States. Magnesium fluoride films were deposited on quartz substrates by evaporation *in vacuo*. Prior to evaporation, the fluoride was fused to a flat spiral filament. The temperature of the fluoride was raised to about 1,400°C, and evaporation was controlled by means of a shutter over the source. Deposition rates varying from 100 Å/min. to 5,400 Å/min. were obtained. Gas pressure was maintained at 0.1 micron Hg during deposition. The dispersion of MgF_2 films was determined interferometrically in the ultraviolet from the wavelengths of the maxima and minima of reflectance and the film thickness. The refractive index of freshly deposited films was 1.375 at 4,200 Å and increased to 1.42 at 2,100 Å. The index increased approximately 1% as the film aged. The index of MgF_2 at the air/film and film/quartz interfaces and the extinction coefficient, were determined from the reflectance and transmittance at normal incidence. The index at the film-substrate interface was consistent with the interferometrically obtained index for the new and aged films. However, the apparent refractive index at the air/film interface depended upon the deposition rate and thickness of the film and coincided with the interferometrically measured index for the thinnest films investigated. The equations for the transmittance and reflectance of absorbing inhomogeneous films are derived. The density of the films was 2.96 g./cm.^{3} and like the thickness, no variation with age could be found.

Article by J. F. Hall *J. Opt. Soc. Amer.* **47**, July 1957 662-665

Dispersion of the Phase Change for Dielectric Multilayers. Application to the Interference Filter — 27/III

See Abstract No.: 146/I

Dielectric Thin Films — 28/III

United Kingdom. This paper is concerned with the properties of thin dielectric films, produced by thermal evaporation *in vacuo*, such as are used in low reflectance coatings, optical filters, etc. The performance of such films often falls short of the theoretical expectations probably because the calculations assume plane parallel-sided, isotopic, homogeneous layers. This investigation is concerned with the properties of single layers of MgF_2, CaF_2, LiF, ZnS and cryolite. The factors affecting the structure and optical properties of such films are listed and it is concluded that, in view of the large number of these, it is not surprising that results on refractive index, stress, etc., show considerable variability. The refractive index of films of all the above types have been measured by (*a*) the Brewster-angle method and (*b*) the measurement of film reflectance. The stress in the films has been measured by the bending of a mica substrate when the films is deposited. MgF_2, LiF and cryolite produce a tensile stress, ZnS a compression stress while the stress in CaF_2 is too small to be measured. The refractive indices of the films are compared with those of the bulk material under various conditions for deposition. The two methods of measurement are not always in agreement and it is concluded that the differences observed in certain cases cannot be ascribed to the use of different methods. The connection between the observed mechanical stress and the structure of the films is discussed.

Article by O. S. Heavens & S. D. Smith *J. Opt. Soc. Amer.* **47**, June 1957 469-472

On Internally Metallising a Betatron Toroid by Vacuum Deposition — 29/III

See Abstract No.: 44/IV

The Development of Electrical Conducting Transparent Coatings for Acrylic Plastic Sheet — 30/III

See Abstract No.: 98/III

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31/III

Light Transmissive Electrically Conducting Article

United States. This patent describes methods of producing light transmissive, electrically conductive coatings for windscreens, aircraft windows, etc., which are adequately durable. The method is to coat the glass substrate with a layer a few molecules thick of an oxide of lead, cadmium, zinc, aluminium, chromium, iron, etc., in order to produce a layer which adheres strongly to the glass and to which the metallic conductive coating will adhere satisfactorily. The oxide layer may be deposited by evaporation of the metallic oxide, by evaporating of the metal followed by oxidation in a gas discharge in the residual air, or by sputtering of the metal in the residual air. The conductive coating consists of a thin layer (not more than 200 Å thick and commonly only 50 Å thick) of gold alloyed with 1% to 5% of one or more of the metals chromium, nickel, manganese, titanium, iron, vanadium or palladium. The conducting layer is deposited by evaporation or sputtering. The addition of the alloying elements serves to improve the durability of the coating without detracting seriously from the desired high light transmission and electrical conductivity. If desired, a protective layer of silica, about 200 Å thick, may be deposited on top of the conductive coating. Data is given for the properties of typical coatings.

Libbey-Owens-Ford Glass Co. *Brit. Pat.* 770,069

32/III

Improvements in the Manufacture of Transparent Conducting Films on Transparent Insulating Backings

United Kingdom. Relates to a method for depositing a thin layer of gold on the surface of a sheet of glass to allow the glass to be heated directly by the passage of current through the gold. Typical application mentioned is for the prevention of mist or ice formation on windscreens. It refers to previous methods using a sprayed or sputtered layer of a semiconducting oxide, or an evaporated film of gold covering a previously deposited oxide layer. In the latter case, the oxide layer improves the adherence and conductivity of the gold. In the method described in this patent, a layer of gold is sandwiched between two layers of metallic oxide (*e.g.* indium oxide), all three layers being evaporated in succession without breaking the vacuum. To do this, the oxide is evaporated from a platinum or platinum-lined boat and the gold from a molybdenum boat or tungsten hairpin. It is recommended to heat the glass to 150°C whilst depositing the oxide and to heat the composite film to 200°C in air for 5 min. In an example quoted, the gold film was calculated to be 32 Å thick and its resistance of 55 ohms per square was reduced to 38 by heating to 200°C. The optical transmission was then between 75 and 80%. Oxides of bismuth, antimony and lead are mentioned as alternative to indium.

A. E. Ennos & Associated Electrical Industries Ltd. *Brit. Pat.* 769,697

33/III

Highly-Conducting Gold Films Prepared by Vacuum Evaporation

United Kingdom. This paper describes an investigation into the properties of gold films deposited on different substrates by vacuum-evaporation methods. A standard evaporation plant was used, the gold being evaporated from a molybdenum foil strip, and the substrate material from platinum or molybdenum foil onto a glass slide. For high conductivity films to be formed a pressure below 5×10^{-5} mm. Hg was essential and the gold had to be evaporated immediately after putting down the substrate material, without breaking the vacuum in between. The gold film was condensed at a rate of 10–20 Å/sec. The thickness of the substrate was estimated by observing interference colours on the glass slides, and the thickness of the gold film by chemical determination of the gold content after all measurements had been made. It was found that substrate materials consisting of the oxides of antimony, indium, bismuth or lead enhanced the conductivity of the gold films. Thus, a 30 Å layer of gold on antimony oxide had a resistance of 50 Ω per square and would dissipate 1 W/cm.² when cooled in oil. A second layer of oxide improved the mechanical properties of the film at the expense of transparency. Measurements of the electrical resistivity of gold films of different thicknesses on a bismuth oxide substrate showed that the resistivity decreased rapidly with thickness from 25 to almost 60 Å (films thinner than 25 Å were unstable) and then more slowly up to the maximum thickness investigated (200 Å). Annealing the films in air reduced the resistance in a consistent manner, depending on the temperature. The maximum annealing temperature was 350°C. Above this, the film began to break up. Films of thicknesses greater than 60 Å, annealed at 350°C, had a resistivity very close to the bulk resistivity of gold. The temperature coefficient of resistivity in the range 20–80°C was found to increase rapidly and almost linearly with thickness up to about 60 Å but this became approximately constant at 1.7×10^{-3} per °C, which is about one half the bulk metal value. Measurement of the Hall coefficient gave values almost the same as that for the bulk metal. Electron diffraction and electron microscope studies of the bismuth oxide and the gold showed that the latter was polycrystalline and non-oriented with an average crystal dimension of 200–300 Å. The oxide substrate was also polycrystalline and non-oriented but with a granularity to the extent of 50–100 Å. This corresponded roughly to the thickness at which the electrical properties of the gold films changed. A comparison with sputtered films of gold on bismuth oxide shows that the evaporated films can be made with a higher conductivity in thinner layers.

Article by A. E. Ennos *Brit. J. Appl. Phys.* 8, March 1957 113-117

34/III

Photoconductivity Speed of Response for High Intensity Excitation in Cadmium Sulphide and Selenide

See Abstract No.: 164/I

35/III

Preparation and Photoconductive Properties of Cadmium Telluride Films

United States. This investigation is concerned with the production of a sensitive photoconducting cell using bimetallic compounds. Cadmium telluride was selected for intensive study because of the large change of resistance with temperature which occurs when a cadmium telluride film cools from 300°C to room temperature

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and because thin films can be easily prepared by evaporation and condensation of the solid. The cadmium and tellurium were purified by zone melting in hydrogen gas and were then combined by heating equal atomic weight proportions in an evacuated tube. Thin films of the material were then deposited by condensation across the short gaps between Aquadag electrodes in Dewar type cells with thin windows. It was found that the film across a $\frac{1}{2}$ mm. gap had a dark resistance of under a megohm immediately after deposition but that the resistance rose as the tube cooled and became 1,000 to 100,000 megohms at room temperature. The resistance, when illuminated with about 2 ft. candles, was 10 to 200 times less. Exposure of the film to iodine vapour reduced the resistance but the photoconductive response also decreased. Doping with indium increased the sensitivity; such cells have dark resistances of several megohms at room temperature increasing to 1,000 and 10,000 megohms when cooled with dry ice and liquid nitrogen respectively. These resistances fall to a fraction of a megohm under low intensity illuminations, whether the cells are cooled or uncooled.

Article by G. G. Kretschmar & L. E. Schilberg *J. Appl. Phys.* **28**, August 1957 865-867

The Reaction of Oxygen with Lead Selenide — 36/III

United Kingdom. Measurements are described of the oxidation of lead selenide, in the form of a powdered single crystal, at various temperatures in the range from room temperature to 280°C. These were aimed at gaining fundamental information about the photoconductive sensitisation of evaporated layers of lead selenide by oxygen. Results obtained indicate that two processes occur during oxidation. In the range of temperature considered the predominating process appears to be the diffusion of oxygen ions to vacant selenium sites in the lattice; this was found to have an activation energy of 17 kcal mol^{-1}. The second process reveals itself only at the highest temperature employed, and is believed to determine the oxidation rate when all or nearly all the selenium vacancies are occupied by oxygen ions. The postulation of two oxidation processes is supported by earlier electrical measurements on lead selenide evaporated layers exposed to oxygen. Theories of photoconductivity in evaporated lead selenide layers sensitised by oxygen are discussed in relation to these experiments.

(*Author*)

Paper by R. H. Jones *Proc. Phys. Soc.* **70B** 1957 1025-1032

Photoconductivity of Thin Films of Arsenic Trisulphide — 37/III

(Reference only)

Letter by H. Schlosser *J. Appl. Phys.* **28**, April 1957 512

Electrical Properties of Arc-Evaporated Carbon Films — 38/III

United States. Uniform thin films of carbon in the thickness range 100 to 2,300 Å have been prepared by arc-evaporation in vacuum. Evaporation rates were about 50 A/sec. The electrical properties of these films suggest that un-annealed arc-evaporated carbon had less long-range order and was, therefore, more truly amorphous in structure than any other form of carbon yet obtained. After the films had been heated to 1,200°K, however, their properties were similar to ordinary fine-particle carbon blacks, apparently as a result of increased ordering. The electrical resistivity of un-annealed films followed Ohm's law, was not photosensitive, and could be well represented in the temperature range 77°K to 300°K by the equation $R=AT^{-b}$, where $b=5$. When the carbon films were heated above room temperature, the resistivity and the exponent were both decreased substantially. After the films had been annealed at 1,200°K, the resistivity was reduced by a factor exceeding 800, and could be best represented, in the temperature range 77°K to 1,200°K, by the equation $R=a-t$. The values of the resistivity, temperature coefficient of resistivity, and thermoelectric power of the annealed films were then more nearly in agreement with the corresponding values for ordinary fine-particle carbon blacks. Other properties and application of the films are discussed.

(*Authors*)

Article by M. D. Blue & G. C. Danielson *J. Appl. Phys.* **28**, May 1957 583-586

Miniature Metallised Lacquer Film Capacitors — 39/III

United States. Describes a new method of constructing capacitors resulting in a volume about one-seventh of that of current types of metallised paper capacitors. The reduction in size is achieved by using a lacquer film (cellulose acetate, is suggested) only one ten-thousandth of an inch thick and evaporating a thin layer of zinc on to one side of the lacquer to act as electrode. To make it possible to handle the lacquer film the latter is prepared by coating in a continuous process on to a strip of paper, from which it is separated by another thin plastic layer to ease the later stripping process. After evaporation of the zinc, the metallised lacquer and the paper with the 'parting layer' are separated and wound on to different rollers. A capacitor is then made by rolling up two strips of metallised lacquer, each strip having one non-metallised margin, the two margins coming at opposite ends of the roll. The two ends are then sprayed with metal to which leads are soldered. The result is a capacitor having a d.c. working voltage of about 50V, more than adequate for most transistor circuits, an insulation figure of merit exceeding 1,000 megohm-microfarads and self-healing properties. An example quotes a power factor for a 1 or 2 microfarad unit of less than 4% at 10 kc/s.

Article by H. G. Wehe *Bell Lab. Rec.* December 1955 441-444

Capacitors having Electrodes Formed of Metallised Dielectric Strips — 40/III

See Abstract No.: 84/I

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41/III

The Metallised Paper Capacitors

See Abstract No.: 82/I

42/III

Chromium Nitride Resistors

See Abstract No.: 87/I

43/III

Electrical Resistances of Thin Metal Films Before and After Artificial Ageing by Heating

See Abstract No.: 96/III

44/III

Study on the Resistivity of Very Thin Gold Films as a Function of the Thickness According to the Nature of the Substrate

France. The high degree of mobility of deposited ^{198}Au films on certain supports can be followed by the measurement of the electric properties of the films. The gold was deposited at 1,550°C from a molybdenum crucible onto a clean glass surface, onto glass coated with bismuth oxide and onto glass coated with magnesium fluoride. Measurements of the conductivity were made after ageing *in vacuo* for 20 minutes. Exponential curves for the resistivity as a function of time and as a function of the equivalent thickness were obtained in measurements for up to 20 minutes and for thicknesses up to 150 Å. The author states that similar results were obtained for silver.

Letter by S. Minn & S. Offret *J. Phys. Radium* **18**, May 1957 357-358

45/III

Electron Mirror Microscopy of Magnetic Domains

See Abstract No.: 60/II

46/III

Magnetic Domain Patterns on Thin Films

United States. Magnetic domain patterns have been observed on evaporated films of iron, cobalt, nickel, and several different alloys of these elements. The films were deposited in the presence of a magnetic field to establish an uniaxial direction of easy magnetisation. It was found that the direction of easy magnetisation. in films of all compositions, could be changed by re-heat-treating them in a magnetic field with a new orientation. This is of special interest because bulk specimens of the elements do not respond to heat treatment in a magnetic field. Nucleation and growth of domains were observed for fields applied at various angles to the uniaxial direction of easy magnetisation in some of the films. Patterns were obtained on films ranging in thickness from approximately 50 Å to 12,000 Å. Domain walls in films ranging from 50 Å to 500 Å in thickness tend to aggregate in pairs.

(*Authors*)

Article by H. J. Williams & R. C. Sherwood *J. Appl. Phys.* **28**, May 1957 548-556

47/III

Domain-Wall Structure in Permalloy Films

United States. Domain-wall structure in Permalloy films in the thickness range 25 to 2,000 Å has been studied by the Bitter technique. A new type of 180° wall has been observed: the main wall is cut at regular intervals by short, right-angle 'cross ties' which terminate in free, single ends. The cross tie period and length are dependent on film thickness, each becoming shorter with decreasing thickness. These patterns can be understood in terms of a new model for the structure of domain walls in thin films of low-anisotropy material. In the conventional model the atomic moments within the wall rotate about an axis perpendicular to the wall. In a thin film such a configuration leads to a large energy contribution from the large demagnetisation factor normal to the film. In the new model, the axis of rotation is itself thought to rotate about the axis of the wall to give a 'corkscrew' configuration of spins and a large decrease in associated magnetostatic energy. Of several experiments which have been performed which support this model, one is briefly described.

(*Authors*)

Article by E. E. Huber Jr. D. O. Smith & J. B. Goodenough *J. Appl. Phys.* **29**, March 1958 294-295

48/III

Ferromagnetic Resonance in Ultra-Thin Films

United States. A ferromagnetic film may be arbitrarily considered as 'ultra-thin' when its thickness is less than 100 Å. It has been possible to observe microwave resonance absorption in Permalloy films down to approximately 15 Å thickness. The magnetisation drops sharply near 60 Å, and ferromagnetism disappears between 8 Å and 12 Å. Line shape and width and g. factor have also been measured.

(*Authors*)

Article by M. H. Seavey Jr. & P. E. Tannenwald *J. Appl. Phys.* **29**, March 1958 292-293

49/III

Steady-State and Pulse Measurement Techniques for Thin Magnetic Films in the vhf-uhf Range

United States. A vhf-uhf bridge consisting of two loops placed symmetrically in a rectangular coaxial cavity has been used to study steady-state and pulse relaxation in thin (1,000 Å) Permalloy films. For steady-state

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measurements a coaxial transformer converts the bridge output from balanced-to-ground to unbalanced-to-ground; measurements from 100 to 1,500 Mc are currently possible. Pulse measurements utilise the ungrounded deflection plates of a wide-band travelling-wave oscilloscope; pulse response to 2 μ sec. can be resolved. Drive-pulse calibration is accomplished unambiguously by using some of the unique switching properties of films which have uniaxial anisotropy.

(*Authors*)

Article by D. O. Smith & G. P. Weiss *J. Appl. Phys.* **29**, March 1958 290-291

Static and Dynamic Behaviour of Thin Permalloy Films

50/III

United States. Thin magnetic films (~1,000 Å) evaporated onto a heated glass substrate in the presence of a d.c. magnetic field develop a uniaxial magnetic anisotropy energy of the form $E_k = K \sin^2 \phi$ with ϕ measured from the deposition field. Such films promise to outperform ferrite cores in digital-computer memory-systems by several orders of magnitude. A detailed static and dynamic theory based on the above anisotropy energy and the Landau-Lifshitz dynamical equation is presented. The static theory contains the unique feature of the coercive force parallel to the easy axis being dependent on a field in the plane of the film and perpendicular to the easy axis. Experimental verification of the theory is given. The dynamic theory considers both steady-state and pulse response. The steady-state solution is well known; the pulse solution must take very careful account of all the important torques acting in a switching experiment, and numerical results are obtained by the use of a digital computer. Experimentally, steady-state resonance is measured from 100 to 10,000 Mc and pulse-switching down to 3 μ sec. Both types of experiment are in good agreement with each other and with the theory.

(*Author*)

Article by D. O. Smith *J. Appl. Phys.* **29**, March 1958 264-273

Reversible Rotation in Magnetic Films

51/III

United States. The magnetisation of Permalloy films of 0.1 to 0.3 μ thickness vacuum-deposited in a magnetic field is reversibly rotated by a pulsed external field applied at right angles to the direction of easy magnetisation. The total angle of rotation is deduced from the amplitude of the detected signal or from the strength of the applied drive field. Reversible rotations of 60° or more are repeated for at least 10^8 cy in some films. An excessive drive field results in a loss of remanent magnetisation after a few rotations. High wall-motion coercivity is a necessary but not sufficient condition for large reversible rotation angles. Films giving a large rotation tend to have open hysteresis loops in the difficult direction.

(*Authors*)

Article by R. M. Sanders & T. D. Rossing *J. Appl. Phys.* **29**, March 1958 288-289

Magnetisation Reversal in Thin Films at Low Fields

52/III

United States. A statistical theory is developed for magnetisation reversed at low fields for thin alloy films showing uniaxial anisotropy. The theory states that reversal takes place by domain wall motion with the number of moving walls as well as the wall velocity controlled by the strength of the reversing field. The theory fits the experimental data for thin evaporated films of Fe, Co and Ni. Brief details are given of the experimental arrangements for measuring the reversal time.

Article by R. L. Conger & F. C. Essig *J. Appl. Phys.* **28**, Aug. 1957 855-858

Flux Reversal in Thin Films of 82% Ni, 18% Fe

53/III

United States. The magnetisation reversal process of iron-nickel films (nominally 82% Ni–18% Fe) deposited in the presence of a magnetic field to a thickness of about 1,000 Å to 4,000 Å have been examined by the application of appropriate fields. Experimental results indicate that at least two different magnetisation reversal mechanisms are effective. The first, characterised by relatively long remagnetisation periods, involves domain-wall movement; the second, characterised by relatively short remagnetisation periods, is consistent with the rotation of the magnetisation in the plane of the film. The threshold for the rotational process is altered by the application of a transverse magnetic field in a manner consistent with a simple energy model.

(*Authors*)

Article by C. D. Olson & A. V. Pohm *J. Appl. Phys.* **29**, March 1958 274-282

Effects of Heat Treatment of Thin Ferromagnetic Films at Intermediate Temperature

54/III

United States. Iron-nickel films deposited *in vacuo* in the presence of a magnetic field have been annealed in an inert environment. It has been established that, when the annealing is performed in an orienting field of 30 oe, initial magnetic properties of these films can be altered provided that the initial temperature is high enough. If a field is applied at right angles to the original preferred direction and in the plane of the film, the magnitude of the magnetic anisotropy can be reduced without changing the preferred direction; the preferred direction of magnetisation can be changed if the anneal temperature is sufficiently high. This last effect confirms the observations of Williams and Sherwood.

(*Author*)

Article by E. N. Mitchell *J. Appl. Phys.* **29** March 1958 286-287

Chemisorption of Gases on Nickel Films I. Kinetic Studies

55/III

See Abstract No.: 173/I

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56/III

Hydrogen Sorption and the Parahydrogen Conversion on Evaporated Nickel Films

See Abstract No.: 172/I

57/III

Thermal Effects in the Chemisorption of Oxygen on Nickel: A Comparative Investigation Using Powders and Evaporated Films

United Kingdom. This note gives the reasons for the differences existing in the measured heats of adsorption of oxygen on nickel powders and on nickel films. Apart from cleanliness, which is an important factor, the question of heat dissipation must be considered. At the instant of oxygen contact, there is intense local heat disturbance at a nickel surface, which may result in oxide formation unless the heat is rapidly dissipated. Under conditions of very bad heat dissipation the nickel may become pyrophoric on account of its high heat of adsorption. A direct determination of the heat of adsorption of oxygen on nickel powder is therefore liable to error. Measurements on thick nickel films on thin glass bases may likewise be inaccurate. The problem with nickel powder has been overcome to some extent by using nitrous oxide which decomposes slowly, liberating oxygen for chemisorption, but the heat of formation of N_2O must be allowed for in the results. A Garner-Veal calorimeter was used in the determination of heat of adsorption of nickel powder, values of which are shown for various volumes of oxygen adsorbed. Saturation corresponded to 2.96 atomic layers of oxygen. Comparative measurements were made on nickel films using the Beeck-type calorimeter. The results show a higher heat of adsorption, saturation occurring when 1.17 atomic layers were adsorbed. The higher heat of adsorption of nickel films may be due in part to a contribution from strain energy. Annealed films might give closer values but the experimental accuracy would be limited by the consequent reduction in surface area. Work on both powders and films is required to give a complete picture.

Note by R. M. Dell D. F. Klemperer & F. S. Stone *J. Phys. Chem.* **60**, Nov. 1956 1586-1588

58/III

The Epitaxial Growth and Oxidation of Nickel, Cobalt and Iron on Rocksalt

See Abstract No.: 158/I

59/III

Reaction Between Oxygen and Hot Gold

See Abstract No.: 21/I

60/III

Oxide Films on Silver at High Temperatures

United Kingdom. Experiments have shown that silver oxide films may exist on the surface of silver at temperatures above 200°C, the temperature at which the oxide in bulk dissociates. The presence of silver oxide on specimens heated at temperatures up to 800°C was demonstrated by the wetting of the treated surfaces by mercury. A preliminary part of the treatment involved heating the specimens of silver *in vacuo*, and then in hydrogen at 450°C to clean and outgas the metal.

Letter by D. E. Davies *Nature* **179**, 22.6.1957 1293-1294

61/III

The Effect of Incident Atomic Velocity on the Structure of Evaporated Silver Films

See Abstract No.: 157/I

62/III

A Note on the Structure of Evaporated Silver Deposits prepared at High Temperatures

United Kingdom. The films have been prepared by evaporation from tungsten filament on to silica plate in an evacuated silica tube. Assembly could be held at temperatures up to 900°C. The silica plate was 0.5 cm. below silver source and vapour stream incident on plate at angles from 90° to about 30°. The rate of deposition at normal incidence was 30–50 Å min.$^{-1}$ (at normal incidence) for total time of 10 min. The deposits were examined by reflection in touch electron diffraction camera with 50–70 kV electrons. There was no orientation in films prepared at 20°C, 340°C and 416°C. At 595°C deposit consisted of large crystals of which a small proportion had a one-dimension orientation about (111) axis. This axis was normal to the substrate even though the incident vapour beam was inclined. Small amount of hexagonal silver was also indicated. At 710°C, only slight amounts of randomly oriented silver remained; predominant orientation with (111) planes parallel to surface but again without azimuthal orientation. (100) and (110) orientations were present in lesser amounts, but no signs of hexagonal silver. Presence of (111) and (311) facets on crystallites indicated. Deposits made at 20°C then annealed *in vacuo* at 600–700°C turned original blue transmission colour of film to milky white and the apparent extent of deposit was reduced. This suggests aggregation and possibly partial evaporation. Heating an initially randomly orientated deposit (formed at 20°C) at 345°C for 40 min. *in vacuo* produced no orientation, but at 610°C and 710°C, strong one-dimensional orientation was present. Orientation was then mainly with (100) planes parallel to substrate, but some (110) and (111) orientation was found. The results are compared with those of other workers.

Research Note by F. H. Herbstein *Proc. Phys. Soc.* **70B**, 1957 251-253

63/III

Study of the Crystallisation and Oxydation of Thin Germanium Layers by Means of Electronic Diffraction

(Reference only)

Article by J. J. Trillat L. Tertian & A. Fourdeux *Le Vide* **11**, July-Aug. 1956 190-193

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Stress Annealing in Vacuum Deposited Copper Films

64/III

United States. An apparatus has been built for the measurement of mechanical stress in vacuum deposited films. A study of the stress changes during pulse annealing has been made with copper films deposited at several substrate temperatures between −150°C and +75°C. The annealing spectrum shows a prominent peak centred near room temperature which matches a resistance annealing peak in similar films and also in cold worked copper. The fall of tensile stress in this region suggests a vacancy mechanism. The theory of Murbach and Wilman for the behaviour of initial stress with temperature is not supported by these results. (*Authors*)

Paper by H. S. Story & R. W. Hoffman *Proc. Phys. Soc.* **70B**, 1957 950-960

Stress in Evaporated Ferromagnetic Films

65/III

United States. Describes studies on thin evaporated films of Ni, annealed in a magnetic field. The films were deposited on mica, annealed at 300°C in a magnetic field of several hundred oersted and then slowly cooled, after which a disc 1.47 cm. in diameter was punched out for experiment. The annealing field was parallel to the plane of the disc, and in a noted direction. Ferromagnetic resonance at K-Band frequencies was then studied for various angles between the direction of the field used to obtain resonance and the direction in the film of the annealing field. The resonant field strength was found to be a maximum when its direction was at 90° to that previously occupied by the annealing field, the peak being greater and sharper when the h.f. currents were in that side of the film remote from the mica substrate (the film thickness was about 10 skin depths, so that the two sides are effectively isolated). The results lead to the conclusion that the film is in a state of tension, the value being about 5×10^9 dyne cm.$^{-2}$ on the side next to the mica and about 8×10^9 on the other side. The stress appears to consist of an isotropic tensile stress of the above values, with a smaller compression superimposed parallel to the easy axis of the film. Possible causes of this are briefly discussed. The results of oscillation magnetometer measurements are also mentioned. These give the saturation magnetisation and the average demagnetisation factor. The saturation figure of 4,780 gauss is in satisfactory agreement with the figure for bulk Ni and with the average calculated for the two sides of the film from the resonance measurements. There is some discussion of the work of Conger and Essig and the suggestion that corrections for stresses might be applied to it.

Article by J. R. Macdonald *Phys. Rev.* **106**, June 1957 890-892

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A New Phase Structure of Molybdenum

66/III

India. The authors have demonstrated that molybdenum deposited *in vacuo* often has a face-centred cubic structure. Previously only body-centred cubic structure of molybdenum had been reported. A pure molybdenum wire was used as filament in the investigations. After initial flashing to remove surface impurities deposition on rock salt and glass substrates was carried out for 10–30 minutes in a vacuum of 10^{-4}–10^{-5} mm. Hg. Electron diffraction examination of the deposits showed their structure to be face-centred except when the substrates were close to the filament when it was body-centred. A detailed account is to follow.

Note by P. S. Aggarwal & A. Goswami *Proc. Phys. Soc.* **70B**, July 1957 708-710

Improvements i.o.r.t. Devices for Vaporising Substances by Means of High Frequency Currents

67/III

United Kingdom. In previously used methods of vaporising metal to act as a getter in a vacuum tube, either a directly heated helical filament, or a metal plate carrying the getter material and heated by high-frequency induction, have been used. The invention describes a method which permits induction heating, while allowing the material to be in the form of a wire held in a helical filament. It also allows the vapour stream to be directed in a desired direction. A piece of thin sheet metal (*e.g.* nickel) is bent to a cylindrical shape, with the ends not quite meeting so that the result is a cylinder with an axial slot running its entire length. The adjacent edges of the slot are joined electrically by the helical filament which is mounted inside the cylinder. When the cylinder is placed in an h.f. field, induced current flows in the circuit consisting of cylinder and helix in series. A piece of wire (*e.g.*) placed in the helix may then be vaporised, and the vapour stream will then only issue from the open ends of the cylinder. It is suggested that it may be confined still further if one end of the cylinder is obstructed by a mica plate.

Philips Electrical Industries Ltd. *Brit. Pat.* 766,769

Method and Apparatus for Coating by Thermal Evaporation

68/III

United States. An apparatus is described which can be used for evaporating larger quantities of material than are usually possible using a filament-type of vapour source for thermal evaporation in high vacuum. The source has been developed for use with materials such as cryolite and zinc sulphide which evaporate by sublimation. A crucible of some refractory such as zirconium oxide or zirconium silicate is filled with a compacted mass of the material to be evaporated, leaving a hole in the centre with an opening at the top. The crucible has a hole in the bottom through which a hairpin filament is passed. The vapour rises through the orifice in the compacted material and can be condensed to make coatings many microns thick on a stationary substrate or alternatively a thin continuous coating on strip material. The advantage of the method is that the heat radiated from the vapour source is very low, so that the temperature of the substrate can be controlled fairly easily. Thick coatings can be deposited because of the large quantity of material available for sublimation.

F. K. Truby & Bausch & Lomb Optical Co. *U.S. Pat.* 2,762,722

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69/III
Note by L. Marton & J. A. Suddeth
Rev. Sci. Instrum. 29, May 1958 440-441

High Vacuum Temperature Gradient Furnace

United States. Thin metal films of approximately equal thickness but different crystallite size have been deposited by evaporation in vacuum onto the cleavage faces of rock-salt crystals held at different temperatures. This has been done by arranging a temperature gradient along a tantalum strip upon which the crystals are mounted. Selected temperature differences can be produced along the strip between the limits 900°C and room temperature.

70/III
W. C. Heraeus G.m.b.H.
Brit. Pat. 766,459

Improvements i.o.r.t. High Vacuum Coating Devices

Germany. The apparatus here described is for carrying out an improved method of vacuum coating foils and papers in continuous strip form. The method has been designed to prevent the outgassing of the materials to be coated in the evaporation or sputtering chamber. The apparatus consists of three separate cylindrical vacuum chambers which are inter-connected by adjustable slit systems through which the foil-like material can pass. The slit systems thus form a vacuum lock, across which a pressure differential may exist. One of the chambers can be used to contain the roll of uncoated foil or paper, the second as the coating chamber and the third to house the take-up spool. A second diagram shows a different arrangement of the chambers using the same principles. The pre-outgassing of the roll of material can be carried out at fore-pump pressure in one chamber, and because of the inter-connecting vacuum lock, the coating chamber can be held at a much lower pressure for carrying out the evaporation. The outgassing can be aided by the use of glow discharge electrodes which can be fitted in one of the chambers. The systems described here have the advantage that the roll of material is not held in the coating chamber where it might suffer deterioration by heat. A magnetic system is also described, whereby the end of the foil can be passed through the slits before starting.

71/III
Article by Anon
Printing News

The Story of Gold II. Gold Foil

United Kingdom. Continuous rolls of real gold foil up to 40 inch. wide are made by G. M. Whiley Ltd. using a vacuum process. Strips of gold (bar, ingot, or 'buttons' recovered from waste) are placed on frames adjacent to moving spools of prepared carrier film. Under high vacuum the gold is atomised on to the carrier and forms a continuous film of gold of the same quality as beaten gold leaf. The 40-inch master roll is then removed from the vacuum chamber and the foil is coated with a heat-sensitive size to make it suitable for hot press stamping. The master roll can be cut to standard widths and lengths. Acetate, viscose or vinyl carrier films are used to suit the material to be decorated, and various shades of gold can be produced. Continuous rolls of gold foil were first produced by the company in 1933 and by 1939 such foil had largely replaced gold leaf for blocking purposes. A recent development is autograph foil or writing foil which makes it possible to write golden signatures and messages on paper or other smooth surfaces. Aluminium is used by the company to make an untarnishable silver-coloured foil, known as Vapco Stamping Foil, which has the brilliance and durability of gold. Beaten gold leaf which may be as thin as 100 millimicrons is also produced.

72/III
M. Roulet
French Pat. 1,129,728

A Method of Vacuum Metallisation and Apparatus for the Same

France. This patent describes a method for the metallisation of small objects of glass, plastics, etc. under vacuum in a continuous series of operations. Essentially, a circular plate with 12 or more positions, carrying the same number of vacuum sealed containers, is made to rotate by a motor with a system of cams or automatic controls which enable the objects to be introduced at the first position into a container. This container is then successively connected in the 2nd, 3rd and 4th positions to vacuum pumps, which lower the pressure inside in stages. At the 5th and 6th etc. positions, heating is applied to a filament of the metal to be vaporised. Cooling under vacuum is realised at positions 8 and 9 etc. Position 12 corresponds to the unloading of the coated objects. The advantage of this method is that by using a series of pumps, higher vacuum may be obtained and by the continuous process it is easy to coat a large number of small objects.

73/III
G. Hoppe & Siemens-Schuckertwerke A.G.
U.S. Pat. 2,768,098

Method and Apparatus for Precipitating Metal from the Vaporous State onto Plates. Particularly for the Production of Selenium-Coated Rectifier Plates

Germany. An improved method of coating a series of plates by evaporation in high vacuum is described. This invention relates particularly to the production of selenium rectifier plates by vaporising the selenium in a vacuum chamber so that the vapour is precipitated on to the prepared rectifier plates. In order to obtain a uniform thickness and crystalline texture over all the plates, they are mounted on a conveyor device which is electrically heated, maintaining the plates at a constant temperature. The final film thickness may be built up after a number of passes over the selenium vapour sources. By this means the plates are held at a fixed temperature over the entire processing, thus ensuring the correct crystalline state of the film and the formation of the barrier layer. A typical apparatus is shown comprising a drum-type of holding jig of polygonal shape on to which the rectifier plates may be mounted easily. An alternative jig is described which uses a conveyor chain travelling at constant speed and carrying magnetic holding devices. The principle aim of the invention is to deposit the selenium in the correct crystalline state for rectification.

74/III

A Vacuum Plant for the Frequency Calibration of Quartz Vibrators

United Kingdom. Discusses the design of a plant for depositing the electrode metal on the thin quartz crystal plates used in h.f. oscillators, filters, etc., in a single operation, including fine adjustment of frequency by

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control of the thickness of metal deposited (for details, see Holland, Electronic Engineering, **24**, (1952), 10) A small chamber is used, the crystal being mounted on an insulator in the lid, so as to provide a connection to an external frequency-measuring circuit. Only one crystal is coated at a time and the plant is designed to give a very short time of loading, pumping and coating, with the possibility of automatic control. To increase pumping speed, the usual high-vacuum valve is omitted, and special measures taken to prevent loss of diffusion pump fluid into the rotary pump and entry of oil vapour into the vacuum chamber. An air-cooled diffusion pump is used, with a patented baffle-type fluid economiser in the backing line—this permits over 3,000 cycles of operation (nearly a fortnight at full production) on one charge of pump fluid. When the system is opened to atmosphere, the heater is switched off to prevent overheating of the oil, while at the same time the cooling fan is stopped to reduce heat losses from the boiler. After 15 sec. exposure to atmosphere, the pressure reaches 0.1 micron in somewhat over one minute. To reduce the amount of oil to be dealt with by the economiser on opening to atmosphere, a magnetic valve isolates the backing pump from the diffusion pump, while a capillary by-pass allows a limited air flow through the diffusion pump to prevent oil mist rising into the chamber. An optical baffle is fitted in the mouth of the diffusion pump to reduce backstreaming and condensation in the chamber is discouraged by keeping this a few degrees hotter than the baffle. The pressure in the chamber is measured by a Philips Gauge connected by a tubulation only $\frac{1}{2}$ inch long. In use, aluminium or gold is evaporated from a tungsten wire or molybdenum boat, the source being pre-heated to dull red heat immediately the pumping cycle starts and then raised quickly to evaporation temperature to allow good control of the evaporation rate. In a typical cycle, 6 sec. is required to raise the pressure to atmospheric, 8 sec. to change the work and about 60–70 sec. to exhaust, *i.e.* an overall time of under 2 min. The chamber is fitted with two lids, so that a new crystal can be loaded while the previous one is in the chamber. A scheme for automatic control of the cycle by an electric clock is suggested.

Article by J. Green L. Holland & B. D. Power *Electronic Engng.* September 1957 440-444

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Improvements i.o.r.t. the Coating of Surfaces with Aluminium by Vacuum Deposition

75/III

United States. An improved method for the continuous vacuum evaporation of aluminium is described. In a continuous coating process, it is desirable that a large source of aluminium should be available, and that the actual area of evaporation should be small, in order to decrease the heat radiation which is transferred to the substrate. This has been achieved in the present invention by maintaining a pool of aluminium at a temperature just above its melting point (800°C). Above this is a high temperature (1,300°C) evaporating zone of comparatively small area, which is fed with aluminium from the pool by means of a wick element. This element must be wettable by molten aluminium, and resistant to attack by it. Preferably, the wick is of carbon coated with a thin film of a carbide or nitride of titanium, zirconium, hafnium, vanadium, columbium and tantalum. Current is passed through the wick element which itself forms the high temperature zone of evaporation. Particular methods of feeding aluminium to the pool and to the wick elements are given. Applications of one or two such sources to a continuous coating process are described.

P. Godley Jr. P. J. Clough R. A. Stauffer National Research Corp. *Brit. Pat.* 762,941

Improvements i.o.r.t. the Vaporisation of Aluminium

76/III

United Kingdom. Aluminium is often evaporated under high vacuum onto plastics, paper and other materials. Evaporation takes place from a base, generally a crucible, plate or rod, and difficulty has been experienced because of the destruction of this base by the action of the aluminium. A method used, for example, is to feed aluminium onto a hot tungsten rod, but this is expensive because of the rapid destruction of the rod. Silicon carbide bonded onto clay, carbon, graphite and carbon coated with zirconium slurry have been tried as base materials but with little success. Much longer life has been achieved using carbides in the form of a solid block at least one eighth of an inch thick instead of as a skin on another material. Of principle interest are the carbides of titanium, zirconium, tantalum and tungsten used in a pure form free from clay binder, metallic and other impurities. The bases, which can be produced by compacting carbide granules under heavy pressure, are porous and can act as their own heating element by conduction through the absorbed molten aluminium. Evaporation can be started using an aluminium wire running through the base, and will cease of its own accord when the aluminium has evaporated out of the pores of the base material leaving it substantially unaffected.

A. Hilliard L. Holland W. Edwards & Co. (London) L'd. & Société le Carbone-Lorraine *Brit. Pat.* 770,751

Coating

77/III

United States. The usual mirror-like appearance of vacuum-evaporated aluminium films is modified to produce a silver-like diffuse reflection for decorative purposes, such as for jewellery, car components, etc. to impart the appearance of chromium plating or the patina of old silver. This effect is achieved by cleaning the article to be coated, and applying a thermosetting resin containing at least 40% of a dispersed solid, preferably ethyl cellulose of particle size less than 10 μ or a finely ground pigment such as zinc chromate or TiO_2. The resin is then cured and vacuum-coated with Al. A final clear resin coating provides protection.

J. Fletcher & National Research Corp. *U.S. Pat.* 2,767,105

Improvements i.o.r.t. Surface Treatment of Metals

78/III

United Kingdom. A process is described for producing a bright coloured and grained finish on metals, which form anodic films, without the use of dyes. The surface of the metal, which may be aluminium, tantalum or zirconium, is etched to bring out the crystal grain boundaries and is then anodised so as to produce a thin oxide film. In the case of tantalum and zirconium the surface appears coloured immediately after the

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anodising process because of the light interference effects set up by the film. In the case of aluminium interference colours develop fully when a thin transparent film of a noble metal or other corrosion-resistant metal is deposited on the anodic film. In each case the colour obtained varies throughout the spectrum according to the potential used in anodising. The etching is carried out in a bath comprising hydrofluoric acid, hydrochloric acid, nitric acid and water. Anodising is effected in a dilute aqueous solution of ammonium borate. For example, pure aluminium having relatively large crystals was etched by immersion for 5 seconds in a bath consisting of 15% hydrofluoric acid, 45% hydrochloric acid, 15% nitric acid and 25% water at ordinary temperature. It was then quickly washed and anodised in a dilute aqueous solution of ammonium borate using a potential of 70V. After washing and drying, a thin film of gold was deposited on the anodised surface by evaporation *in vacuo*. Interference colours of purple then became visible, the surface having a granular and scintillating appearance. Potentials of 5–250V may be used for anodising according to the colour required. Zirconium and tantalum may be similarly etched and anodised. The anodised film produced is very resistant to many reagents with the exception of chlorides. A final coat of clear varnish, such as 'Zapon', may be applied to the treated surface. Aluminium specimens having large crystals which show a definite grain structure are readily obtained. Zirconium and tantalum, however, usually have small crystals and the crystal grain boundaries are not so evident on etching.

A. Charlesbury
J. J. Polling &
Nat. Res.
Dev. Corp.
Brit. Pat.
763,336

79/III

Vacuum Deposited Films of Nickel-Chromium Alloy

United Kingdom. The preparation and properties of stable films of vacuum deposited nickel-chromium alloy are described. Properties discussed include the film alloy composition, the relationship between film thickness and resistance (Ω/square), the temperature coefficient of resistance, grain size in the film (electron micrographs), mechanical failure of stressed films, optical transmission as a function of film thickness, fundamental noise and electrical breakdown under low and high frequency conditions. Graphs summarise the data and micrographs illustrate grain growth, breakdown due to stresses in the film and electrical breakdown in micro-wave attenuators.

(*Authors*)

Article by
R. H. Alderson
& F. Ashworth
Brit. J. Appl. Phys.
8, May 1957
205-210

80/III

Simultaneous Evaporation of Pt and C for Possible Use in High Resolution Shadow-Casting for the Electron Microscope

United Kingdom. The resolution obtainable with shadowed specimens in the electron microscope is limited by aggregation of the shadowing material. A non-crystalline material of high atomic number is required. Such a shadowing material has been deposited by evaporation from a resistance heated contact between rods of a sintered platinum/carbon mixture containing about 85% Pt by weight. Very sharp shadows have been produced using this technique.

Article by
D. E. Bradley
Nature
181, 29.3.1958
875-877

81/III

Evaporation of Boron

United States. An apparatus that has proved satisfactory for the evaporation of boron is described. Boron is loaded on a carbon support stand and heated by a tungsten filament placed just above this. Of the boron thus evaporated 1% passes through the hole in a surrounding molybdenum shield. For the size of evaporation apparatus described—external diameter $\frac{3}{4}$ inch, height 1 inch—an 8 mg. boron charge is used. The power required to maintain an evaporation rate of 8 mg./hour, using a 25-mil tungsten filament is 250 W. Operational temperature of the shield is about 1,200°C, that of the filament 2,300°C, and between 2,200–2,300°C for the boron.

Note by
H. A. Hill
Rev. Sci. Instrum.
27, Dec. 1956
1086

82/III

Improved Technique for the Evaporation of Boron

United States. Thin uniform coatings of boron are required for parallel-plate ionisation chambers and proportional counters. The technique described here involved resistance heating in vacuum of a carbon boat in which a small charge of boron is placed. Spectroscopic-grade carbon rod (diameter $\frac{3}{8}$ inch) is used for the boat and this fits closely into two molybdenum sleeves to give low-resistance electrical contact at operating temperatures. The depression in the carbon rod which holds the boron (0.1 g.) is the only point of high resistance in the system and therefore reaches m.p. temperature of boron (2,300°C) without causing molybdenum evaporation. A layer of boron carbide is formed in the depression which also effectively eliminates carbon evaporation. With such an arrangement 35 consecutive boron evaporations have been made using 150 A at 5 V. A 0.025 mg./cm.^{2} layer is deposited to an estimated uniformity of 1%.

Note by
G. J. Safford
Rev. Sci. Instrum.
27, Nov. 1956
972

83/III

The Uses of High Vacuum Evaporation for the Metallising of Textiles

See Abstract No.: 45/I

84/III

In Combination

United Kingdom. The metallising of glass is involved in the manufacture of reflecting surfaces, electrically conducting surfaces, and glass-to-metal seals, as well as for reinforcement purposes. A recent application is the coating of glass fibres. It is found that a zinc coating is greatly strengthened by the addition to the zinc of 0.5–7.0% of titanium, zirconium, hafnium, vanadium, niobium or tantalum. The resultant yarn is flexible and has a strongly adherent coating.

Note by
Anon.
Metal Industry
20.1.1956
42

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Radiant Heat Reflecting Clothing 85/III

See Abstract No.: 256/I

Improvements i.o.r.t. the Production of Metallised Effects on Textiles 86/III

Switzerland. A decorative irridescent appearance is given to textile material of open structure by metallising one side by vacuum deposition of a metal vapour. The fabric must have clearly visible interstices between the yarns and these interstices should preferably amount to at least 25% of the total area of the fabric. When a material of such a loose structure, for example muslin or voile, is treated in this way the metal deposited on one side is invisible when the other side is viewed in a direction perpendicular to its surface, but becomes 'locally' visible as an irridescence or sheen when the fabric is viewed obliquely. The fabric may consist of cellulosic, animal or synthetic fibres and may be dyed, printed, parchmentised or treated with a transparent finish. Aluminium is the metal preferred but other metals such as copper, silver and gold are also suitable. If desired, two different metals, for example copper and gold, may be deposited successively on the fabric. In one example described, mercerised cotton muslin, dyed navy blue, having a yarn count of 90/82.5 per inch and a British yarn count 80/120, is aluminised on one side in a high vacuum chamber. When the non-treated side of this fabric is viewed obliquely, its aluminised side becomes visible as a silvery sheen. Metallised textiles of the type described are used mainly for ladies' wear and for curtains.

Heberlein & Co. A.G. *Brit. Pat.* 774,223

Improvements i.a.r.t. Reflective Fabrics 87/III

See Abstract No.: 257/I

Improvements i.o.r.t. Flexible, Porous, Metallised Sheet Material 88/III

United Kingdom. This patent refers to the method of metallising a fabric in such a way that the fabric may breathe. The method consists in applying, by means of a spraying apparatus or printing roller, an adhesive, such as 'Bostik' 1768 diluted with ethyl acetate, in such a way as to avoid filling the interstices of the fabric. The fabric is then metallised by either a transfer method or by vacuum evaporation. Experiments show that, if a given volume of air under a given pressure will pass through unit area of untreated viscose rayon fabric in one second, then it will take two seconds when the fabric is metallised as described above and twelve seconds when the adhesive is spread on the rayon by a doctor knife.

Imperial Chemical Industries Ltd. R. D. Brace & R. W. Maxwell *Brit. Pat.* 767,714

A Replica Technique for Particle Surfaces 89/III

See Abstract No.: 76/II

Uses of Carbon Replicas in Electron Microscope 90/III

See Abstract No.: 70/II

31 — CATHODIC SPUTTERING — 31

Electroluminescence 91/III

See Abstract No.: 117/I

Theory of the Sputtering Process 92/III

United States. Gives a theory of the sputtering process, using mathematical methods similar to those used to study the diffusion of neutrons in solids. Physically, the theory postulates that a beam of ions, mass M_i, is incident on the surface of a metal and penetrates,'cooling' as a result of collisions into thermal equilibrium with the metal lattice. As a result of the collisions, some of the metal atoms, mass M_j, are set into motion within the lattice, and themselves suffer collisions with other stationary, lattice atoms. Some acquire sufficient energy to escape from the surface. Collisions between moving particles are assumed to be negligibly infrequent. The system is taken to consist of two distributions of moving atoms, the 'incident' and the 'struck', which interact with the stationary lattice particles. From the two distribution functions, the sputtering ratio (number of metal atoms sputtered per incident ion) is determined. To obtain the equations, each of the distributions is first split, the first into the 'beam', consisting of all those particles which, although they have entered the metal, have not yet made a collision and still have their incident energy (assumed the same for all ions), and also those which have collided and lost some energy, and the second distribution into 'lattice particles', being metal atoms which have been struck, and the 'metal'—particles of both types in thermal equilibrium. The Boltzmann equation is then used to derive for each type of particle an equation relating the distribution of particles to energy, penetration within the metal and direction cosine to the normal.

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Energy is expressed as the logarithm of the ratio of the energy of an incident ion to that of the particle considered. Parameters used in the equations are the 'trapping parameter', involving the scattering and trapping cross-sections, the ratio of the mean-free paths for the two types of particles, the mass ratio (M_i/M_j) and the threshold energy. The sputtering ratio is finally evaluated by integrating the flux of metal atoms for all values of energy between the incident and threshold energies, giving an expression involving the above parameters. The results are then compared with some experimental data, due to Keywell, for the systems He-Pb, He-Ag, Ne-Ag, A-Pb, A-Ag, A-Cu, Kr-Ag, Kr-Cu, covering a range of mass ratios. Choosing the trapping parameter and mean-free path ratio by trial and error, gave curves which agreed reasonably well with the experimental curves except for the smallest mass ratios, in which cases it seemed that the assumption of a single value of the trapping parameter for all values of beam energy was not satisfactory.

Article by D. E. Harrison *Phys. Rev.* **102**, June 1956 1473-1480

93/III

Sputtering of Surfaces by Positive Ion Beams of Low Energy

United States. Silver, germanium, and germanium-silicon alloy surfaces have been bombarded with the inert gas ions Ne^+, A^+, Kr^+, and Xe^+, at energies between 30 and 400 eV. Primary beam intensities between one and ten μ/cm.^{2} were obtained in an oscillating electron source of the Heil type. The secondary particles sputtered off, neutrals as well as positive and negative ions, were identified and recorded in a 180° mass spectrometer of 5.5 inch. radius. They are partly background gases adsorbed on the surface, partly atoms and molecules characteristic of the sample. Among the latter particles, neutrals were about 100 times more abundant than those emitted as positive ions. Rather low yields (about one sample atom per 100 incident ions with 400-eV energy) and relatively high threshold energies (40–50 eV for an angle of 30° between beam and surface) are believed to be due to surface contamination. Retarding potential measurements showed that over 80% of the particles sputtered have initial energies of less than 5 eV. After bombardment, the Ge and Ge-Si surfaces showed oval-shaped hillocks, possibly due to appreciable carbon concentrations in the samples studied. If the effects of surface contamination can be reduced, this method promises to be useful in the analysis of solid surfaces.

(*Author*)

Article by R. E. Honig *J. Appl. Phys.* **29**, March 1958 549-555

94/III

Differential Sputtering of Grain Boundaries in Polycrystalline Tin

India. Refers to previous reports that properties of grain boundaries depend on differences in orientation of adjoining grains, and to the authors' previous observations of differential etching during cathodic sputtering of Sn. In the further work described, high-purity white tin was etched by slow sputtering in argon at 0.1 mm. Hg, using 500 V and a gap of 1.2 cm. A micrograph (× 100) shows the intersection of three boundaries. From the measured dihedral angles at the intersection, the relative boundary energies are calculated and hence the relative dislocation densities. It can be seen from the photograph that the boundary with the highest dislocation energy has sputtered to the greatest extent, which is said to support the 'transition lattice' theory of the grain boundary.

Letter by P. G. Deo & B. D. Sharma *Nature* **178**, 13.10.1956 812-813

95/III

The Effect of Thin Films of Gold on the Mechanical Properties of Cadmium Single Crystals

United Kingdom. Thin films of oxides, hydroxides or metals have been shown to increase the stress at which plastic deformation starts by an amount which cannot be explained by the strength of the films themselves. Most experiments have been on creep, showing the effect of oxides and hydroxides. This paper records an investigation into the effect of thin films of gold on the stress-strain curves of cadmium crystals. The crystals were grown in an Andrade-Roscoe furnace and extended in a rigid frame machine with a Polanyi beam and photographic recording. The gold films were deposited by sputtering. It was found that thin gold films increased the stress at which plastic deformation started by about 6 g. wt. mm.$^{-2}$. The increase was independent of film thickness for films of 1,500–24,000 Å. The nature of the effect of the film upon the stress-strain curves depended upon the orientation of the crystals.

(*Authors*)

Paper by F. R. Lipsett & R. King *Proc. Phys. Soc.* **70B**, 1957 608-620

96/III

Electrical Resistance of Thin Metal Films Before and After Artificial Ageing by Heating

United States. The electrical resistances of thin films of 24 metals, deposited on glass substrates (near room temperature) by evaporation, sputtering, or electroplating, has been measured before and after artificial ageing by heating *in vacuo*. The films varied in thickness between 100 and 1,500 Å. Evaporation was carried out at 2×10^{-5} mm. Hg and at distances of 5–10 cm. Sputtering was carried out at a pressure of about 3×10^{-2} mm. Hg in an unstated gas, using currents of about 20 mA and voltages approaching 3,750 volts. For electroplating standard solutions were used with currents of 1 mA per sq. cm. and the film was deposited on a thin base-film produced by evaporation or sputtering. Thickness determinations were made by geometric calculation and weighing supplemented by occasional interferometric checks. Electrode connections to the films were made *via* triangular copper-foil leads using indium as the solder. It was found necessary to carry out the ageing procedure in an infra-red vacuum oven to avoid oxidation and other chemical efforts. Graphs are given showing the variation of resistance (in ohms per square) with film thickness before and after ageing. It was found that reductions in resistance of 25 to 50% as a result of ageing were commonly obtained. Evaporated films were reduced from about $2\rho_b$ to $1.3\rho_b$ (ρ_b = resistivity of the bulk metal) and sputtered films

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from 4–10ρ_b to 1.5–1.8ρ_b. A preferred ageing temperature, specific to each metal but influenced by film thickness, is quoted. This temperature, which is in reasonable agreement with that obtained by electron microscopy and diffraction studies of the films, is of the same order as the temperature of recrystallisation of the metal. It is suggested that thin metal films, as usually deposited, are in a state of strain, not associated with the bulk metal as it crystallises from the melt. The application of heat energy to the film promotes the removal of these strains, the release of occluded and adsorbed gas, and the growth of the crystallites of the film. These effects reduce the electrical resistance of films of 1,000 Å thickness to a value usually in the range 25–75% above that of the bulk metal. It is emphasised that, when the electrical properties (and also the light transmission properties) of thin films are important, the films should be given a suitable ageing treatment.

Article by R. B. Belser *J. Appl. Phys.* **28**, January 1957 109-116

Light Transmissive Electrically Conducting Article

97/III

See Abstract No.: 31/III

The Development of Electrical Conducting Transparent Coatings for Acrylic Plastic Sheet

98/III

United States. Methods are discussed for the preparation of transparent, conductive films on both acrylic and glass surfaces. The theory of the structure of such films is presented as well as a description of techniques involved in the preparation. Techniques described are sputtering of metallic cadmium under conditions such as to form the oxide, and the preparation of thin continuous films of silica and titania.

(*Authors*)

Article by G. A. Dalin & R. W. Freedman *W.A.D.C. Technical Report* 53-378

Metal Films

99/III

United Kingdom. Pilots can be protected from the effects of solar radiation by depositing a thin gold film on the cockpit cover. Such a film can have an optical transparency of 75%, yet reflect infra-red radiation well. Electrical loading up to 1 watt per cm.^{2} is possible for de-icing. The film is formed by sputtering gold onto the cover in a 36-inch. vacuum plant. Good adhesion, a low electrical resistance and high light transmission are obtained if the gold film is sandwiched between two layers of bismuth oxide. The vacuum plant contains emitting cathodes and an offset vapour source so that multilayer films can be produced in one cycle. Even deposition is achieved by rotating the work. The mechanism by which a metal deposit attaches itself to a glass base is not clearly understood. Metal films deposited in low pressure atmospheres of oxygen, sulphur or selenium were found to be much more adherent than a film deposited in low-pressure air. Such films can be used as resistance thermometers at temperatures up to the softening point of the glass base. Contact between the leads and the film is achieved by fusing gold electrodes onto the glass and depositing the film over them. Leads can be connected to silver terminals which are fused onto the glass and make contact with the electrodes.

Note by Anon. *Engineering* 20.12.1957

Method of Making Transparent Conducting Films by Cathode Sputtering

100/III

United Kingdom. After reviewing processes previously proposed for making thin transparent electrically conducting films of metallic oxides which were later reduced to a lower oxide, *e.g.* stannic oxide. The author proposes a process by which surfaces which can withstand temperatures up to 400°C may be coated with a partially oxidised film of tin or indium by cathode sputtering of the parent metal in an argon atmosphere containing traces of air. The sputtering conditions must be such as to give a sputtered layer which has a distinctive colour, a yellow to brown coating. This is subsequently heated in air to 350°C for five minutes. The colour disappears during heating leaving a transparent coating the resistance of which makes it suitable for use as a windscreen heater pad, a hot stage for optical microscopy or a variable resistance for use in volume controls in electronic equipment.

J. S. Preston & National Research Development Corp. *U.S. Pat.* 2,769,778

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Electron Diffraction at Electrolytically Polished Surfaces Cleaned by Cathodic Sputtering

101/III

Germany. An electron diffraction camera is described which is fitted with a cathodic sputtering device for the cleaning of metal surfaces. In this apparatus electro-polished single crystals of copper, brass and iron as well as cleaved surfaces of zinc are investigated. The cathodic sputtering apparatus is employed to remove the surface film generated during the electro-polishing process of the crystals. In this manner it was possible to observe a copper surface cleaned by cathodic sputtering, while admitting air, whereupon a film of copper oxide could be seen. This is not the case where cathodic sputtering is not used. In the case of cleaved zinc surfaces, a very thin layer of zinc oxide can be observed after the sputtering process but, in agreement with previous experience, this film does not show the same morphological structure as zinc, *i.e.* its lattice constant is different from that of zinc.

Article by A. Ladage *Z. Phys.* **144**, No. 4, 1956 354-372

Improvements i.o.r.t. Etching Metals

102/III

United Kingdom. It is well known that the technique of cathode sputtering involves the disintegration of a cathode surface under positive ion bombardment, and that this technique can be used for etching the surface of a metal to reveal the crystalline structure. The mechanism by which an etch pattern is formed is not fully understood; it might be due to selective sputtering over different parts of the cathode surface, or to a migration

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of atoms into preferred positions on the surface. It is also possible that an artificial pattern could be built up on the surface by condensation of sputtered material, returned by collision with gas molecules. If the sputtering pressure is lowered sufficiently, then the material returned to the cathode can be reduced to negligible proportions, thus avoiding the risk of an artificial pattern. The sputtering pressure may be lowered from the normal (0.01 mm. to 0.1 mm.) to at least 1 micron by the use of a magnetic field to sustain the glow discharge. The magnetic field can be produced conveniently by means of an electro-magnet, situated so that the field is normal to the cathode surface. In order to maintain the symmetry of the discharge, an auxiliary cathode of aluminium (or some other metal having a low sputtering rate) is fitted. In order to prevent the cathode from overheating, which might cause changes in the structure, cooling should be provided. One form of a suitable apparatus is described, and a diagram is given. Using this apparatus a very good etch pattern has been obtained with metals and alloys which have hitherto been difficult to etch by chemical methods. It is thought that the successful results are due to the fact that the lower working gas pressure reduces the risk of a contaminating layer being formed, whilst also increasing the effective sputtering rate.

L. Holland & W. Edwards & Co. (London) Ltd. *Brit. Pat.* 779,347

103/III

Etching of Germanium Crystals by Ion Bombardment

United States. A study is made of the etch effects produced by sputtering germanium crystals and bicrystals under normal incident low-energy (100 ev) Hg^+-ion bombardment in a low pressure plasma (1 micron gas pressure). The surfaces show preferentially etched pits which readily reveal the crystal orientation. These pits become more abundant at lower target temperature and truncated at higher target temperature or at higher energy of the bombarding ions. A sequence of micrographs from the same surface area reveals that these pits cannot be correlated with the pits which are persistently developed by CP–4. A 6° tilt boundary however always develops into a sharply indented furrow just as was the case with a CP–4 etchant.

(*Author*)

Article by G. K. Wehner *J. Appl. Phys.* 29, February 1958 217-221

104/III

X-Ray Radiation Hazard Using Vacuum Deposition Plant

United Kingdom. Reference is made to a report by Strachan and Harris that a sputtering plant operated at 8–10 kV can give rise to dangerous x-radiation. This conclusion was based on observations made on a sputtering plant in which the ions were formed in an auxiliary discharge, the pressure in the sputtering chamber itself being lower than normal. The x-rays were generated by electrons released from the target bombarding the walls of the glass envelope. The article reports on tests carried out on a normal two-electrode sputtering plant using a cold cathode. In such an apparatus the pressure is higher and hence the emitted electrons will not attain an energy equivalent to the ion accelerating potential difference. The tests showed that no x-ray exposure was detectable after 8 hours on test films placed on the outside of the Pyrex bell jar, of wall thickness 8 mm., in which a discharge was operated at 18–20 kV and 20 mA.

Letter by L. Holland *Nature* 180, 13.7.1957 89

33 — IMPREGNATION — 33

105/III

Large Vacuum Impregnator for the Pakistan Railway

(Reference only)

Note by Anon. *Engineer* 203,3.5.1957 701

106/III

Vacuum Impregnation of Telegraph Poles

Germany. The bark is stripped off the lower 3 ft. of the poles and this section is enveloped in a plastic bag which contains the preservative, a solution of 1% copper sulphate and 3% ammonia having been found to give satisfactory results. The mouth of the bag is closed with a strap, and a cupping-glass fitted with a Plexiglass pump is employed. The plastic bags may be re-used 30 times, and the method is much quicker than the traditional Boucherie method.

Note by Anon. *Timber Technology* August 1957

107/III

Industrial Power Capacitors

See Abstract No.: 83/I

108/III

Impulse Voltage Strength of Oil-Immersed Insulation

United Kingdom. Describes investigations into the behaviour of oil-immersed insulation when subjected to steep-fronted unidirectional impulse voltages, such as arise from lightning strokes or switching surges. In the experiments, the standard 1/50 μ sec. positive waveform was used. The method of application was to start with 70% of the estimated breakdown voltage and increase in 5% steps until breakdown occurred. The transients were recorded by c.r.o. and camera. Specimens were tested in an oil tank 12 ft. in diameter and 12 ft.

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deep containing class B oil which could be heated to 90°C. The first experiments were made with bare electrodes, rod to rod, rod to plane and hemisphere to plane being the combinations tested. For various spacings the mean values of flashover voltage are recorded as a function of the time lag to flashover. Tests were then made on solid insulation immersed in oil, 10 inch square samples being used according to B.S.231. Data is given of the mean impulse puncture voltage of Grade 3 elephantide sheet in various thicknesses, along with the corresponding figures for breakdown at 50 c/s. An empirical relation found between the mean impulse puncture voltage and the thickness is:—$V = 5.9d^{0.79}$. (d being in mils and V in kV_{kp}). Similar data for resin-bonded leads to $V = d^{0.84}$. Experiments are then described on the breakdown between paper-wrapped conductors, oil-immersed, with and without vacuum impregnation of the wrapped conductors. In one series of tests, the only pre-treatment was air drying at 110°C for 72 h. followed by immersion in class B oil at 20°C for 48 h. The results are expressed by:—$V = 5d^{0.65}$, where d is now the diametrical thickness in mils. Similar tests in which the air drying was followed by vacuum impregnation in class B oil at 100°C and 1 mm. Hg for 72 h. gave improved figures of:—$V = 7.1d^{0.72}$. Another series in which the paper-wrapped conductors were held apart by elephantide spacers also showed considerable improvements due to vacuum impregnation. It is concluded that there is little to choose between impregnation pressures of 1 mm. and 9 mm. Hg. The article concludes with the results of tests on surface breakdown made on elephantide strips 16 inch. × 1 inch × $\frac{1}{4}$ inch, with electrodes either of copper strip on edge or of 20 s.w.g. copper wire wrapped round the strips. Impulse tracking voltages up to 600 kV_{pk} are recorded with type 2 pressboard. Failure resulted in deep carbonised failure of the top lamination. It is reported that synthetic resin bonded paper has a higher impulse strength with a relatively low resin content. The effect of polarity of the impulses is said to be slight and it is suggested that work is needed on breakdown with combinations of normal and tangential stresses.

Article by H. F. Jones *Elect. Energy* December 1956 103-108

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36 — DRYING — 36

A Graphical Method for the Calculation of Cooler-Condensers

109/III

See Abstract No.: 100/I

Vacuum Unit for Dehydrating Coolers

110/III

France. It is well known that water in a cooling system is always a cause of trouble. One-tenth of a drop of water in a system filled with Freon–12 is sufficient to cause a stoppage. Hence the necessity for complete dehydration. The two principal methods are: (*1*) circulation of hot air and (*2*) desiccation by vacuum. The first method is cumbersome, large quantities of air being necessary, and it is also unreliable. The second system is far more practical; the vacuum pumping system being mobile, it can be brought to the installation to be dehydrated. In practice, the necessary pressure is 0.5 to 1 mm. Hg at normal temperature. The Edwards dehydrating group comprises an air injection pump having a limit of 0.02 mm. Hg and a pumping speed of 32 l/min. This group can also be used to remove the humidity from silica gel desiccating filters. It can also be used to fill cylinders with refrigerating liquid without risking contamination by air or other refrigerants.

Article by Anon. *R. Gen. Froid* **34**, Jan. 1957 87-88

The Design and Performance of Small Climbing-Film Evaporators

111/III

United Kingdom. The climbing-film evaporator consists of a vertical tube heated from the outside. Liquid is fed in at the bottom and warms to boiling point as it rises. A froth of liquid and vapour is formed, and higher up the tube vapour bubbles almost fill the space, so that the liquid is carried upward as a thin film. At the top, the tube discharges into a separator, the vapour passing to a condenser and the liquid into a receiver. The first part of the report lists various design equations and considerations regarding tube dimensions etc., and gives details of a particular design. In the second part of the report entrainment and corrosion are discussed. Of the various entrainment separators tried a column packed with glass wool gave the best performance, and improved the characteristics of the evaporator to an acceptable level. Corrosion of the stainless steel tube was not excessive.

Report by D. Pepper *A.E.R.E. Rep. No.* CE/R 1779 April 1956

A Simple Rotary Film Evaporator

112/III

New Zealand. A way of eliminating difficulties in connecting the rotating flask of a rotating film evaporator to a stationary vacuum line is described. The flask is attached *via* a standard taper joint to a glass tube of internal diameter 5–6 mm. The upper end of the tube is connected to the vacuum system by pressure tubing. A fixed metal collar acts as a bearing for the tube, and holds it at about 15° to the horizontal, the bottom of the flask resting in a bath. A flanged metal wheel is cemented to the tube, and a length of cord or light chain passes round it. The upper end of the cord is attached to a fixed support *via* a spring or elastic band, and the lower end is fixed to a collar on a crankshaft driven by a motor at 55 r.p.m. Rotation of the crankshaft causes vertical movement of the cord, resulting in rotation of the flask forward and backward through about 360°. This rotation is completely accommodated by the vacuum line. By way of example, at a pressure of 1 mm. Hg and solution temperature of 20°C (bath temperature 21.5°C) evaporation was greater than 50 ml./h.

Article by B. B. Marsh & P. M. Nottingham *J. Sci. Instrum.* **33**, Nov. 1956 446-447

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Article by H. Mueller *Chem. Ing. Tech.* **30**, 1958 133-137

High-Frequency Vacuum Drying

Germany. On the basis of preliminary experiments it is shown that dielectric drying *in vacuo* offers advantages in respect of both the temperature requirement and the drying time. Experiments in a high-frequency condenser field and in a dipole radiation field were carried out, in each case under conditions most unfavourable for high-frequency drying. In the high-frequency condenser field considerable saving in time was achieved, even when operating at low temperature.

(*Author*)

114/III

Article by F. Fixari W. Comley & G. K. Viall *Chem. Engng. Prog.* 53, March 1957 110, 112, 115

Continuous Vacuum Dehydration Reduces Cost

United States. A new continuous-flow vacuum dryer is described. Present applications include soluble coffee, citrus bioflavinoid compounds and orange and grapefruit crystals. Pilot plant runs on tomato paste, milk, food supplements and chemical products are taking place. A diagram of the plant is given. A roller feeds material to be dried in the form of a liquid concentrate on to a 4 ft. wide stainless steel belt moving in a vacuum chamber around two large roller drums which are $7\frac{3}{4}$ ft. in diameter and 35 ft. apart. Puffing takes place when the material is deposited on the belt and this facilitates subsequent drying and improves the appearance and solubility of the final product. Initial heatings achieved by several separately controlled radiant elements heating through the belt. The heating produces a porous structure in the material and brings it up to the temperature of the main heating drum, which operates between 140 and 300°F. 80 to 90 per cent of the drying occurs on this drum, further radiant heat being then applied to complete the dehydration and to partially collapse the puffed material. Quick cooling, which is beneficial after drying, is achieved on the second drum into which coolant is piped through on rotary joints in the anti-static bearing. An oscillating "Doctor" blade removes the dried material from the belt. The latter is passed by a screw to one of two receivers which are alternatively filled and removed from the machine by means of a vacuum lock. The cooling down serves to cool the belt in readiness for the next application of material. Cycle times vary with material from 15 seconds to a few minutes. The temperatures are made as high as possible to attain a large pressure difference between the product vapour pressure and the operating pressure. The latter can be in the range from 500 microns to 10 mm. Hg. Most products are dried at from 1 to 6 mm. Hg.

115/III

Article by K. Brinkmann & M. Beyer *Elektrotech. Z.* 24, 11.12.1956 881-884

Developments in the Dielectric Drying of High-Voltage Cables

(Reference only)

37 — METALLURGICAL PROCESSES — 37

116/III

The Application of Vacuum in Metallurgy

See Abstract No.: 52/I

117/III

Article by R. R. Giler *Steel* 15.7.57 108-110

Vacuum Heat Treating Takes Hold

United States. Some examples of industrial cold-wall vacuum furnaces which incorporate radiation shielding are described. The cold wall unit of the Mallory-Sharon Titanium Corp. is designed to vacuum anneal and degas titanium and features a car-type loading which enables the load to be placed either directly on the car or suspended from a fixture attached to the car. Finished sheets, for example, can be suspended individually to facilitate degassing and maintain reasonable flatness. The unit has a usable charge space of 4 × 4 × 12 ft. Cooling to room temperature is assisted by filling the furnace with argon which can be circulated internally by a fan. The low heat storage and low impedance of the furnace make it possible to use ejector-type pumps and the cycle is relatively short compared with that of a double pumped vacuum furnace. For equal loads of titanium the degassing time of this type of furnace is one-half to one-third that of a standard hot retort. Cold-wall furnaces are of particular use where fast cooling is required as in brazing special types of stainless steel. A unit operated by the Air Research Mfg. Co., for special vacuum brazing operations, has a charge space of 2 × 2 × 4 ft. and a maximum operating temperature of 2,150°F. The vacuum pumping system consists of a rotary Roots-type blower pump backed by a 130 cfm mechanical pump. With this system there is no danger of oil back-streaming into the furnace or of the vacuum becoming high enough to cause vaporisation of the brazing material. The furnace has a low thermal inertia and cooling can be speeded up by circulating inert gas. Cold-wall furnaces are also used for heat-treating large parts where a hot retort of suitable size is not practical. A furnace being designed at Westinghouse, for example, is large enough to braze a complete aircraft wing. Since the furnace wall is cold it is possible to extend members through it to compress the work being processed. Unlike hot retort furnaces, which are not suitable for use above 2,100°F, cold wall units can be used up to 5,000°F. The trend is towards general purpose types of furnaces with applications ranging from stress relieving at 1,100°F to brazing at 2,100°F in the same furnace. A new type of furnace proposed for semi-continuous annealing at high temperatures has a loading and unloading mechanism and an evacuated cooling chamber on both sides of the vacuum furnace. Charges are prepared, loaded and removed from each side in turn. The chief problems in continuous units are the operation and maintenance of the inner valves which are difficult to reach and which must be leak-proof.

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Recent Developments in Vacuum Heat Treating Furnaces

118/III

United States. Vacuum furnaces suitable for heat-treating titanium are described. Standard metallurgical atmospheres (composed of CO, CO_2, N_2, H_2) cannot be used with titanium since they react with the metal. Protection is only afforded by heating in vacuum or inert gas and contamination takes place again as soon as the metal is brought into air for working. Titanium with an oxidised surface regains its brightness when vacuum annealed but in the process oxygen diffuses into the metal lowering its mechanical properties. Scale can be removed by mechanical means, or by pickling in which case hydrogen diffuses into the metal. Hydrogen lowers the ductility of titanium and specifications usually demand a hydrogen content less than 150 parts per million (by weight). Where large amounts of hydrogen are present the metal must either be scrapped or refined in vacuum by a combined annealing and degassing treatment. For refining on an experimental or small production scale the work can be held in an alloy container connected to a pumping system. The container, or retort, is welded to make it vacuum-tight and placed in a furnace which may be similar to the standard bell or pit-type furnace. In some furnaces, an additional rough vacuum is maintained between the retort and the furnace shell by a single-stage mechanical pump. This eliminates stresses on the retort other than those due to its own weight and repeated heating and cooling. A disadvantage of these hot retorts is that they are liable to distort and it is then difficult to maintain a vacuum seal. For semi-continuous operation a furnace of at least three stages is required. The work is loaded into an entrance chamber which is evacuated before the gate valve of the heating chamber is opened. After the heating operation the metal is transferred through a second gate valve into an evacuated, water-jacketted cooling chamber. A separate pumping system is required for each stage, a large pumping system being necessary for the heating chamber where a large volume of gas is released. A new type of furnace is the Kold-Retort furnace in which the heating element is placed inside the retort. Radiation shields of sheet metal provide thermal insulation and eliminate the need for refractory linings. The charge is placed on a tray that rests on the framework containing the heating elements. The furnace shell is made of mild steel, the largest having a maximum thickness of $\frac{1}{2}$-inch. Molybdenum or Hipernik are the metals generally used for structural work. The presence of chromium must be avoided since it starts to distil out of alloys at pressures of about 1 μ and at temperatures above 1,000°C. Deposition of this chromium on the heating element supports might cause arc-over from the elements to ground. Cooling is effected by a water jacket in the larger or by oil in the smaller units. To increase the cooling rate argon or helium can be bled into the furnace and circulated by fans. The furnace makes it possible to supply heat to any particular region of the charge by a suitable distribution of the heating elements. A further advantage is its low impedance to gas flow which is important in annealing where high gas loads are involved. At present Kold-Retort furnaces range in size from small development units operating at 3,200°F to brazing units with a charge-space 50 ft. long by 20 ft. wide. A recent application of vacuum furnaces is for the bright annealing of stainless steel and the brazing of honeycomb structures for aircraft. In the future the vacuum furnace may replace the protective atmosphere furnace for the heat treatment of metal.

Article by R. R. Giler *Indust. Heating* **23**, Oct. 1956 2038-2040 2042, 2044 2046, 2048 2050

Heat Treating Precision Parts

119/III

See Abstract No.: 56/I

The Successful Use of Vacuum Heat Treating Methods with Atmosphere-Reactive Metals

120/III

United States. The combined action of heat and vacuum provides the most satisfactory way of age hardening, annealing and degassing of certain metals which do not require quenching in order to bring about their best physical properties. Pioneer industrial work in this field was done by the Hamilton Watch Company from 1940 onwards. Elinvar alloy used in hairspring and Dynavar mainspring alloy required the use of vacuum for successful age hardening without oxidation. Impurities in an inert-atmosphere furnace could cause oxidation affecting the functioning of springs with a cross section 0.0007 inch × 0.003 inch mirror finished to an accuracy of 0.000005 inch. Springs treated in vacuum come out of the oven brighter than when they entered it after treatment for 4 hours at 1,200°F followed by 4–6 hours cooling *in vacuo*. Vacuum annealing has merits for the treatment of 420–F stainless steel and for the newly developed age-hardened steel 17–7PH. The metals titanium and zirconium can only be cold worked successfully if they are gas-free. Titanium has a high strength to weight ratio at temperatures up to 850°F, a high resistance to corrosion and it does not flavour or colour foods. It can be annealed, degassed and rolled to a perfectly flat foil 0.0022 inch thick which is nevertheless strong enough to be used to make shutter blades for highspeed cameras. Vacuum-treated zirconium can be cold worked. The metal has unusual corrosion resistance even to nitric acid and hot phosphoric acid. Since it does not absorb neutrons it can be used as a liner for atomic energy reactors of the thermal neutron type. The use of vacuum will increase very rapidly as the demands for these metals increase and their costs become competitive with commercial alloys.

Article by H. L. Hovis *Metal Treating* Nov.-Dec. 1956 2-4

Vacuum Heat Treatment Furnace Makes Titanium Parts Ductile

121/III

United States. Titanium metal alloys become brittle, weak and difficult to machine through absorption of hydrogen. Millions of dollars worth of hydrogen-embrittled scrap have been salvaged by treatment in a vacuum heat-treating furnace built by the Pacific Scientific Co. of Los Angeles, U.S.A. The unit consists of an electrically-heated furnace placed inside a vacuum-tight nickel-chromium alloy retort which is connected to a high speed diffusion pump backed off by a roughing pump. Vacuum of .2 microns can be achieved and furnace tempera-

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tures above 2,000°F are possible. Controls enable the temperature to be held to close limits and there are facilities for inert atmosphere operation. Under typical conditions titanium sheet scrap is degassed for at least six hours at 1,300°F, and then cooled for four hours to 400°F at which point the vacuum is released. Degassing of thicker sections takes longer. Tests showed that the tear and notch tensile properties of titanium sheet were increased almost four times by vacuum degassing. Before degassing 91% of the failure in tear tests were of the brittle type. After degassing all the tests showed ductile type failure.

Note by Anon. *Modern Metals* 13, March 1957 74

122/III

Vacuum Melted Alloys for Forged Parts

United States. The advantages obtained by vacuum melting and developments in the use of vacuum-melted metals are considered. The consumable electrode process has been used for some years for melting metals such as titanium, zirconium and molybdenum and has more recently been applied to Ni-base superalloys, Fe-base superalloys and to stainless and other steels. An advantage of the method is the reduction of the oxides which occur in air-melted material and impair the workability of the metal. This reduction results from the dissociation of the less stable compounds under the low pressures and high temperatures involved in arc melting. The more stable compounds, such as titanium nitrides, which have a lower specific gravity than the parent metal, float out and this also helps the cleaning process. The breakdown of oxides and other compounds is accompanied by the evacuation of the liberated oxygen. Dissolved gases are also forced out of solution and evacuated. A further advantage of melting in a cold mould by the consumable electrode process is that segregation is prevented since only a relatively small pool of molten metal exists in the mould at one time and migration of elements is thus restricted. Consequently the mechanical properties are approximately the same throughout the ingot. Vacuum melting improves the high temperature properties of metals and affords the possibility of increasing the endurance strength by raising the titanium content of titanium-bearing precipitation hardening alloys, while still maintaining good hot workability. Increasing the titanium content in A–286 alloy improves endurance strength at 10^6 cycles and 1,200°F from 37,000–45,000 p.s.i. to above 55,000 p.s.i. Consumable electrode vacuum melted metals are available as forging billets, extruded shapes, bars, rod, tubing, sheet, strip and wire. A disadvantage of the consumable electrode process is the difficulty of controlling composition by making alloying additions. Close composition control can be maintained by induction melting, but with this process segregation is likely to occur on freezing. A combination of the two vacuum processes using induction melting to produce an electrode for subsequent remelting by the consumable electrode process is to be examined with electrodes weighing 1,000 lbs. In America, alloys remelted under vacuum by the consumable electrode process cost about 50 cents to 1 dollar per pound more than corresponding air-melted materials. Their applications include special purpose springs, heavily loaded bearings, highly polished rolls and components requiring high tensile strength. An indication of the growing production of vacuum melted alloys is the increase of the Allegheny Ludlum monthly ingot output from 250,000 pounds in December 1955 to 1,000,000 pounds by the end of 1956.

Article by W. W. Dyrkacz & E. E. Reynolds *Product Engng.* Feb. 1957 143-145

123/III

Refractories and Mould Materials for Vacuum Melting and Casting

See Abstract No.: 109/II

124/III

Coating a Carbonaceous Surface with Rhenium

See Abstract No.: 218/I

125/III

High Temperature Vacuum Furnace

United States. Description of a commercial furnace to be used for sintering.

Note by Anon. *Engineer* 203, 26.4.1957 655

126/III

Vacuum Melting and Casting Furnaces

United States. Description of commercial furnaces.

Note by Anon. *Engineer* 204, 18.10.1957 578

127/III

Method and Apparatus for Melting Metal under Vacuum

United States. A feature of the vacuum melting and casting furnace described is a charging chamber which permits the main and alloying charges to be added in a pre-determined order without breaking the vacuum in the furnace. Inside the furnace shell is a crucible which can be tilted from outside the furnace to pour the molten metal into ingot moulds mounted on a turntable. The top of the furnace is provided with a charging opening on which is mounted a large valve housing, sealed with a pair of circular, vertically-spaced valve gates.

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The central part of the housing is enlarged and extends on one side into a wide casing into which the gates can be withdrawn to open the valve. The gates are supported by inclined toggle links pivotally connected to them and to a piston rod that extends into a cylinder supported in the casing. When fluid pressure is applied to the cylinder the valve gates are pressed vertically against circular gaskets on their seats by means of the piston rod and toggle links, and the valve is thus closed. When fluid pressure is withdrawn from the cylinder the piston rod pulls on the links and the gates are pulled towards each other by coil springs. As the rod continues to pull, the gates are drawn into the casing to open the valve. Arcuate shoulders between the valve housing and casing prevent the gates from sliding on the gaskets and wearing them. The bottom of an air-tight charging chamber is detachably connected with the valve, for example by bolts that connect the flanged lower end of the chamber to the top of an extension of the valve. The top of the charging chamber is sealed by a cap and carries an electric motor connected to a pulley inside the chamber. This pulley supports the charges which are lowered through the open valve into the crucible. When all the charges have been added the valve is closed and air is admitted to the charging bell which is then removed and reloaded through its open lower end. When charged, it is replaced over the furnace and evacuated. The valve is then opened and further charges can be added to the crucible. One method of loading the charging chamber is to place the main charge in a large sheet metal bucket which is hung from the pulley by fusible links. Suspended from the bottom of the can is a string of small batches of alloying elements which may be carried in pails connected by fusible links. When the charge already in the crucible has been melted the first addition can be made by lowering the assembly until the lowest pail is in the crucible. When the fusible link melts the remaining charges can be raised into the charging chamber until the next addition is required. When the last small addition has been made the large bucket can be left down in the furnace to pre-heat the main charge for the next melting. As soon as pouring has been completed this main charge is lowered into the crucible and whilst it is melting the charging chamber is removed and reloaded with the additives and the next main charge. Instead of suspending the alloying agents from the bottom of the next main charge they can be hung inside the large bucket that holds the main charge to which they are to be added. In this case the lowering mechanism is arranged so that the string of small pails can be lifted out of the large bucket when the latter is lowered into the crucible.

J. D. Nisbet & Universal-Cyclops Steel Corp. *U.S. Pat.* 2,788,270

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Automatic Feed Casting Apparatus

128/III

United States. The apparatus described is intended for casting or sintering under vacuum, and may also be used as a degassing or reacting apparatus for treating materials prior to casting. Molten metal for casting is contained in an induction-heated crucible housed in a vacuum chamber. This crucible is supplied from one or more feed crucibles which are also induction-heated and maintained under vacuum. The connecting pipe between the feed and casting chambers is evacuated and is fitted with means of controlling the flow of metal, *e.g.* with a plunger. Metal is added to the feed crucibles through a valve. The moulds are supported one above the other in a tube which extends vertically through the housing and crucible of the casting chamber. Openings in the length of tube which passes through the crucible permit metal to flow in and fill the moulds which are automatically pushed downwards through the tube. The lower end of the tube is cooled to solidify the metal in the moulds. Feed and withdrawal of moulds is automatically controlled by two rotary members situated one at each end of the tube and driven in synchronism by means of a shaft. Passages are arranged in the upper member so that one passage, filled with fresh moulds, is aligned vertically with the mouth of the tube while another is aligned with a hole in the cover plate to allow for reloading. The passage aligned with the tube is evacuated through a port in the cover plate and the moulds are pushed down into the tube by a plunger which is evacuated and is preferably fitted with a liquid seal. In a similar way filled moulds are pushed from the bottom of the tube into an evacuated passage in the lower member. When the passage is full the member is rotated until the passage is aligned with holes in the cover plate which permit ejection by a plunger. The vacuum within the tube serves to degas and deoxidise the mould cavities and ensure sound castings without air pockets. Metal or other material may be degassed, alloyed or otherwise pre-treated in the feed chambers before passing to the casting chamber. The feed chambers may also serve as reaction chambers. For example chemicals for making synthetic mica may be fluorinated under pressure and the resulting material then passed into the casting chamber. Powdered metal briquettes can be fed into and withdrawn from the tube in the same way as moulds, and can be sintered by a high-frequency induction coil surrounding a portion of the tube. Sintering under vacuum in this way is said to improve the electrical conductivity of the briquettes.

J. B. Brennan *U.S. Pat.* 2,787,817

A Small Vacuum Induction Furnace

129/III

United Kingdom. The furnace deals with 300 g. of high melting point alloys and is designed for vacuum melting and pouring. The vacuum tank is an 11 inch. tube of $\frac{3}{16}$ inch mild steel outside diameter $6\frac{1}{4}$ inch., with flanges at each end. The lower flange sits on the oil baffle of an Edwards Type 403 diffusion pump and the upper flange, of the same diameter as the lower, is fitted with two location pegs to position the lid which carries an O-ring seal. Coaxial leads to the r.f. coil surrounding the crucible enter *via* a Wilson seal and the coils are made interchangeable by the use of $\frac{1}{4}$ inch petrol unions. The earthed outer conductor consists of two coaxial copper tubes with sealed ends and cooling water flows through the gap between them. The inner conductor is a $\frac{1}{4}$-inch copper rod surrounded by Araldite, the outer end being wax-sealed. Each heater coil has a diameter 4 mm. greater than the crucible to allow for a 2 mm. coating of alumina cement on the latter. The cement is baked at 1,500°C before use. In melting iron and chromium to form alloys, the chromium must be added gradually to the molten iron as otherwise the chromium forms a solid arch over the molten iron. Additions are made from a hopper built in the furnace lid and divided into six sectors. On rotation of the hopper the contents of one sector fall through a slot into a chute which is then operated by an external lever. A Pyrex glass observation window fitted with a shutter on the vacuum side is included in the lid plate. The melt is cast into a split

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cylindrical copper mould mounted on a vertical rod and adjustable in position. The pump system is protected from metal splash by a copper gauge. The crucible is loaded with iron and the hopper with chromium pellets. Pressure is lowered to 10^{-4} mm. in 3 mins. and r.f. power applied gradually. The iron melts after approximately 10 mins. Chromium is then added with the observation window shuttered. Pressure rises to several mm. Hg with vigorous outgassing. After pumping to 10^{-4} mm. Hg more chromium is added and after adding all the chromium the r.f. power is increased until the vapour pressure of the melt exceeds 10^{-3} mm. Hg. Pouring is then carried out. During melting intense ionisation occurs in the vacuum space but since the generator supply was adequate no steps were taken to reduce it.

Article by J. C. Matthews *J. Sci. Instrum.* 34, February 1957 62-63

130/III

A Laboratory Electron Bombardment Furnace

United Kingdom. This article describes a furnace for the melting of refractory metals. As in previous practice, the ingot is melted on a water-cooled copper hearth, to avoid the difficulties due to reactions between melt and crucible. In contrast to previous practice, however, electron bombardment in high vacuum is used instead of a low-pressure argon arc. This is claimed to give better evaporation of volatile impurities from the melt. The furnace, which can melt ingots up to $3 \times \frac{1}{2} \times \frac{1}{2}$ inch, is made in three sections. The lower section—made from a 2 inch. diameter stainless steel cylinder—carries the hearth, which is the earthed anode. Above it is a glass cylinder 12 inch. high and 12 inch. in diameter, which insulates the top plate carrying the cathode assembly and serves as a window. The whole assembly is mounted on top of the baffle-valve of a 9 inch. diffusion pump. The copper hearth, which is bored out to give channels for the cooling water, is mounted on a steel piston which slides in a double O-ring seal in a side tube projecting from the lower section of the chamber, the sliding motion allowing movement of the ingot relative to the electron beam. The cathode assembly projects downwards into the chamber from a modified Wilson seal; it can be moved vertically in this seal and also laterally to a limited extent by toggle action. The actual emitter is a flat spiral tungsten filament, operated at up to 12 V, 20 A and about 1 inch above the ingot. Accelerating potentials of up to 9.5 kV are used, the power unit having a capacity of 750 mA. Pressures below 10^{-4} mm. Hg are quoted, with a pumping speed of 900 l/s, the leak rate being of the order of 301 μ/h. Examples given of the use of the furnace are in the purification of niobium and uranium. In the case of niobium, it is reported that after four periods of about 5 minutes melting (the sample being inverted after each melting so that the same side was not always in contact with the hearth and therefore solid) the hardness had been reduced from 120 to 40 V.P.N. and the oxygen content from 0.047 to 0.001%. Some uncertainty about the removal of nitrogen was eliminated by an experiment with a sample which had been deliberately contaminated with nitrogen.

Article by P. G. England & H. N. Jones *J. Sci. Instrum.* 35, Feb. 1958 66

131/III

Apparatus for Vacuum Casting of Uranium

See Abstract No.: 216/I

132/III

A High-Vacuum Casting Furnace for Plutonium

See Abstract No.: 217/I

133/III

Electric Furnace for Production of Metals

United States. In the arc furnace described each electrode is carried by a massive, water-cooled support designed to eliminate any possibility of water or other cooling fluid leaking into the furnace and causing an explosion. The furnace is of the skull type and is intended primarily for melting titanium and other reactive metals. Melting and pouring are carried out in a vacuum-tight enclosure in an atmosphere of low-pressure argon. The electrode support should be made of a metal such as copper which has a high thermal conductivity. It is preferably forged from a single billet so as to form a solid mass long enough to extend to the outside of the vacuum chamber through a suitable seal. A passage down the axis of the support contains a pipe which circulates the cooling water. The end of the passage is at least 5 inches from the electrode and the amount of copper between the water and the electrode is sufficient to maintain a temperature drop of less than about 500°C along the electrode support. This ensures that the temperature at the electrode is below the safe operating temperature of the support. The diameter of the lower end of the support is at least two inches greater than the diameter of the water passage which should be about one or two inches. The bottom wall of the passage should be at least one inch thick. Several heat shields are arranged close to the bottom of each support to minimise radiation of heat from the arc to the support. Each support can be raised or lowered a predetermined amount, *e.g.* by pneumatic means, to enable the electrode to clear a heat shield fitted over the crucible and to permit tilting the crucible for pouring. If the electrode failed completely during operation of the furnace, the support would be burned away by the arc until the arc length became too great to be supported by the voltage. This would occur before the arc ruptured the end of the water passage but the brilliant green colour characteristic of arcing to copper should provide a warning before the trouble reached this stage. An additional warning could be given by a temperature-sensing device in the cooling water supply to each individual electrode. To facilitate heat removal from the bottom of the crucible a titanium stud which extends from the skull through the bottom of the crucible, is connected to a massive conductor which also serves as a ground return for the electrodes. When the crucible is in the melting position the conductor contacts a stationary matching cooling block, preferably of copper, which extends through the vacuum chamber and which is fitted with a passage for cooling water at its lower end.

J. L. Ham & National Research Corp. *U.S. Pat.* 2,789,152

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Heat Reservoir and Electrode for Production of Metals in Skull Type Furnace

134/III

United States. A 'skull' furnace is generally used for melting reactive metals like titanium under vacuum or in an inert atmosphere. Basically it is a vacuum chamber containing a crucible above which there are one or more electrodes. The crucible consists of a shallow bowl which is made of a refractory substance and has an inner lining composed of an electrically conducting substance having a high melting point. Graphite is often used. After evacuation to a few millimetres of mercury and the introduction of inert gas if required, the metal placed in the crucible is melted by heat generated by an arc struck between the metal and the electrodes above it. The furnace derives its name from a layer or 'skull' of unmolten metal which remains at the end to separate the molten region at the top of the crucible from the graphite layer at the bottom. During the melting a considerable amount of heat is generated at the electrodes and in the past it has generally been the practice to cool them with circulating water to prevent them from melting. However, in such systems there has always been the danger that the cooling fluid would react with the molten metal, resulting in burn-out, explosion and danger of injury to operating personnel and equipment. This danger has been overcome by replacing the cooling fluids by a heat absorbing reservoir or 'heat sink' consisting of a massive piece of copper into which the electrodes are set. Experience has shown that it takes about 25 Kw.hr. power input into the furnace to melt one pound of titanium and that roughly 20 per cent of this power is dissipated at the electrodes. It is therefore possible to calculate the amount of heat that has to be absorbed by the reservoir so that the electrodes shall not rise above a certain temperature during the course of the melting process. In practice the mass of copper needed is affected by such factors as the shape of the reservoir, the heat loss through the skull and the efficiency with which the reservoir is screened from radiant heat leaving the molten metal. A safe installation employs a mass of copper ten times as large as that of the titanium to be melted.

P. J. Clough
J. L. Ham
D. I. Sinizer &
National Research Corp.
U.S. Pat. 2,789,150
April, 1957

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Arc Melting Furnace

135/III

United Kingdom. In the arc-furnace described a number of small charges, for example test buttons of refractory metals such as titanium, can be melted consecutively without opening the furnace between each melt. The need to re-establish the vacuum or protective atmosphere between each melt is thus avoided, and cooling of the test buttons can proceed in the furnace during the melting of the following charges. A further advantage is that the atmosphere within the furnace may be 'gettered' either by using the first melted charge to absorb all gaseous contaminants, or by other means. The remaining charges may then be melted without increase in their oxygen and nitrogen content and are therefore considered to give a more representative sample of the material under test than do buttons produced in other furnaces. The hearth comprises a rotatable, water-cooled plate provided with a number of cups or depressions, which serve as melting stations, equally spaced from each other and at the same distance from the centre of the hearth. The hearth and its cooling chamber are supported on a centrally disposed hollow shaft which can be rotated by means of a handwheel, and which carries the inlet and outlet pipes of the water supply. The hearth is housed within an air-tight casing which incorporates a turret situated in line with the path of rotation of the cups. On the upper surface of the turret a water-cooled electrode is supported upon detachable flexible bellows which permit vertical and angular movement of the electrode whilst maintaining gas-tightness. In operation, the hearth is rotated to bring the cup containing the first charge to be melted into alignment with the electrode which is then manipulated to strike the arc. The progress of melting is observed through a window in the turret, and on completion the arc is broken and the hearth is again rotated to bring the next charge below the electrode.

P. J. Robinson &
Imperial Chemical Industries Ltd.
Brit. Pat. 789,895

Improvements i.o.r.t. Arc-Melting Furnaces

136/III

United Kingdom. British Patent No. 737,043 described a vacuum arc melting furnace for melting reactive metals such as titanium, zirconium, molybdenum, thorium, etc. The arc is struck between the metal and a non-consumable electrode which can be moved and which enters the furnace through a gas-tight seal. A scraper surrounding the electrode is intended to prevent the fouling of the seal by metallic deposits and condensed volatile matter which have been deposited on the electrode. In practice some of these deposits have been found to build up on the scraper ring in a loosely coherent manner instead of falling back into the melting crucible. The built-up material can bridge the gap between the electrode stem and the furnace lid and cause an electrical breakdown when the electrode and lid are of opposite polarity. This has been overcome by fitting a sleeve of insulating material to the steel tube mounting the scrape ring.

Imperial Chemical Industries Ltd.
Brit. Pat. 787,264

Melting Metals in Vacuum-Arc Furnaces

137/III

Germany. Factors influencing volatilisation and degassification of the metal, in particular of titanium, and the effect of low pressure gases on the electric arc are discussed. Good mixing of the molten metal is necessary to avoid temperature differences in the bath, to separate alloy components and to bring dissolved gases to the surface and hence aid their removal. Mixing is more effective in a high frequency furnace which has a vertical stirring effect than in a d.c. arc furnace in which the bath is mixed as in a centrifuge. When carrying out degassification and deoxidation *in vacuo* it is necessary to provide an artificial, low dynamic pressure of the metal vapour which should be sufficient to counteract metal volatilisation but low enough to permit removal of permanent gases. When smelting volatile metals such as chromium and manganese in the vacuum arc furnace this effect is achieved by admitting rare gas to adjust the pressure. Diffusion of gas to the surface is easier when the melt has a large surface area. A disadvantage of the vacuum arc furnace with a consumable electrode is that the free bath surface area is small and a dynamic gas pressure is easily set up between the electrode and the bath or crucible. Smelting occurs too rapidly and equilibrium may not be reached between

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the hydrogen dissolved in the metal and the atmosphere. An open electrode arc furnace with two consumable electrodes, developed by L. Weiss, should be more suitable for degassification than smelting in a long, cylindrical crucible. The most favourable arc conditions for rapid melting occur near the critical pressure at which the arc would become a glow discharge. Tests in which a titanium anode was melted under decreasing pressures of argon showed that no melting took place during a glow discharge, and the cathode spot was observed to creep up along the copper electrode and stay at an activated point where it caused rapid destruction of the copper and burned a hole in the water jacket. Pressure-voltage curves for an arc between a tungsten tip and a hot tungsten cathode show a sharp minimum at about 0.6 mm. Hg and 70 V in a hydrogen atmosphere and a broad minimum between 0.25 and 5 mm. Hg at 25 V with an argon atmosphere. Argon is not, therefore, a suitable gas for avoiding a glow discharge. Helium behaves similarly to hydrogen. A minimum is also observed with a glow discharge in a titanium arc furnace. The removal of hydrogen from solid titanium depends on the surface condition and is hindered by the oxide film which is produced in a vacuum at 790°C. Heating above 850°C causes diffusion of the oxide into the metal and facilitates movement of the hydrogen. Titanium rod, 12.5 mm. thick and containing 43 ppm. hydrogen, was found to lose 77% of the hydrogen in less than 4 hr. when heated to 815°C in a high vacuum, which indicates a rapid diffusion of hydrogen. Titanium sponge prepared by reduction with Mg and distillation at 950°C in a pressure of less than 1 μ can have a hydrogen content of less than 15 ppm. When a sponge electrode is used with a water-cooled copper electrode, some of the gaseous Mg and $MgCl_2$ from the hot electrode tip condense on the copper wall and electrode and are subsequently entrapped by the rising melt as melting proceeds. The salts are then overheated and evolve as gas which carries away some of the metal. This collects above the bath in the form of a 'spray crown' rich in chlorides. Spray drops entrapped by the bath make it necessary to remove the surface of the solidified metal by machining. High chloride pressure in the electrode tip also causes spatter of the sponge.

Article by W. J. Kroll *Metal Treatment and Drop Forging* **24**, April 1957 162-168

138/III

Improvements i.o.r.t. Electric Arc Furnaces

United States. An electric arc furnace for the melting of titanium or similar metals is described in a number of modified forms. The furnace achieves results better than those of the consumable-electrode type without the necessity of producing a compressed and welded titanium-sponge anode. In principle, the furnace employs an arc spread over a large area, which melts titanium introduced in powdered form. The anode consists either of conventional material *e.g.* copper, or of titanium. The use of a high vacuum, say, 5 mm. Hg encourages the arc to spread and avoids the need for a protective atmosphere. The arc is directed onto the molten titanium surface by a cylindrical arc-shield or baffle, which prevents the cathode spots of the arc from spreading to the walls of the container, even at very low pressures. Diagrams of the type of furnace employed and three modified forms are included. The melting pot is made of copper or similar metal, and has a piston-type base by means of which the molten surface of the ingot may be kept at a fixed distance from the anode. The anode is a coil or helix of copper tubing surrounding a small starting anode of titanium. Melting is begun by striking an arc between a starting lump of titanium in the melting pot and the starting anode. The latter is then lifted away, and the arc is formed between the main anode and the molten titanium. A current of 2,000 A or greater is required to melt the powdered titanium as it is fed into the melting pot from a hopper. The arc shield or baffle surrounding the anode extends to below the anode, but remains just clear of the upper molten surface of the ingot. The baffle, melting pot walls and copper anode are directly water-cooled. When the melting operating is completed, the ingot is removed from the lower end of the enclosure, and the powdered titanium replenished in the hopper. In a modified form of furnace, the anode has the shape of an inverted funnel so that the arc level is at variable heights. The entire anode assembly is raised to maintain a constant separation from the molten surface. The starting-function is fulfilled by a small replacement consumable starting anode of titanium. Another modification employs three water-cooled anodes arranged in a triangle, whilst an external magnetic means of shielding the walls is used. This necessitates the use of non-magnetisable melting pot walls and cooling-jackets. A fourth modification employs as a main anode an uncooled solid block of titanium or piece of compressed titanium sponge. This runs fairly hot, but is not melted and introduces no impurities. The furnaces described were designed to produce ingots larger than 12 inch. in diameter, and good titanium may be produced with only one melting.

Westinghouse Electric International Co. *Brit. Pat.* 769,053

139/III

Improvements i.o.r.t. the Arc-Melting of High Melting Point Metals or Alloys

United Kingdom. In the apparatus described a coherent but incompletely melted metal mass, produced by arc-melting in an upper crucible, is lowered into a second crucible where it serves as the consumable electrode for a second melting operation. An initial charge of divided metal, such as titanium, is fed from a hopper into the first crucible and an arc is struck between the charge and a solid consumable electrode. The latter may consist of a forged bar or of units consolidated from divided metal and welded or otherwise joined into a suitable length. When the arc has been struck divided metal is fed at as fast a rate as possible consistent with the production of a coherent mass in the crucible. The maximum feed rate is advantageous in that it limits the amount of electrode consumed in the operation. When the mass has been built up to a suitable height in the crucible, the feed is stopped and a molten pool is allowed to form at the top of the mass. The electrode is dipped into the pool so that it fuses together with the mass and serves as a stem by which the metal can be supported in the lower chamber. In order to permit movement of the mass into the second chamber, the water-cooled base-plate of the upper crucible can be lowered by a retracting mechanism to fit into a seating aperture and form the base plate of the second melting crucible. Particles of metal which may fall on the seating surfaces are removed by a brush attached to the retracting mechanism just below the base plate. When the mass is in the required position to form the electrode in the lower chamber an initial charge is fed into the crucible from the hopper and an arc is struck between the charge and the mass. The latter is then

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melted into the crucible with or without the addition of an extra charge. The lower crucible must be longer than the upper so that the top of the mass is clear of the upper crucible when in the second position. It must also be of larger diameter so that the charge may fall freely between the mass and the walls of the second crucible. If desired the upper crucible may be tapered to facilitate the downward movement of the mass. Several upper crucibles may be used in conjunction with a single lower crucible of sufficiently large diameter. Before use the whole apparatus is evacuated by a vacuum pump after which it may be refilled with inert gas if required.

Imperial Chemical Industries Ltd. *Brit. Pat.* 769,502

Improvements i.o.r.t. Consumable Electrodes Used in the Melting of High Melting Point Metals or Alloys

140/III

United Kingdom. A continuous, consumable electrode which can be fed into an arc-melting furnace may be formed by compacting the raw metal in particulate form onto a continuous length of wire or strip. The use of welding for joining separate sections is eliminated. Sponge or powdered metal, for example titanium or molybdenum, is fed vertically down from a hopper into a die chamber through the centre of which passes a continuous element of wire or strip. The charge is compacted around this element by means of power-operated punch members which have their inner faces shaped as required. The shape of the punch members is preferably such that they produce an electrode with a tapered upper end. The compacted segment is withdrawn through an aperture in the base of the die chamber until only the tapered end remains in the chamber. This tapered end serves as an insert around which the next segment can be compacted thereby improving the rigidity and continuity of the continuous electrode. Heat as well as pressure may be applied in the compacting process if desired. Electrodes of any required length can be built up for subsequent feeding into an arc furnace, but it is preferable to feed the electrode directly into the furnace as it is formed. In this case the driven rolls which withdraw successive segments from the die chamber also serve to feed the resulting electrode into the melting crucible. The compacting process is carried out whilst the electrode is stationary in the furnace. The length of each segment is governed by the effective variation in arc length or a multiple thereof and the rate of withdrawal is controlled according to the speed of melting. If required, a number of compacting dies may be used to form a number of electrodes which are fed simultaneously into the same melting furnace. The wire or strip should preferably provide a mechanical key for the compacted metal and serrated wire, perforated, embossed or dimpled strip or expanded metal strip may be used. The wire or strip may have the same composition as the metal to be melted or it may be composed of or contain alloying additions.

R. Atkins & Imperial Chemical Industries Ltd. *Brit. Pat.* 770,448

Multiple Crucible Arc Melting Furnace

141/III

United States. In the arc furnace described buttons of seven different refractory alloys can be melted in one charging cycle. The main body of the furnace is of stainless steel and consists of a double-wall cylinder with an inner diameter of 6.755 inch. and a height of 7.93 inch. A ring welded to the top and a flange welded to the bottom of the walls enclose the space between them to form a water jacket. Viewing windows and ports for water and vacuum and gas connections are provided through the walls. The bottom flange is screwed to a copper pad, in which are machined seven crucibles, 1-inch dia. by 0.5 inch deep, spaced equally around a 3.62-inch. dia. The pad also forms the top of a water chamber. The furnace cover is a stainless steel plate, 7 inch. dia. by 0.625 inch thick. It carries Leucite electrode supports positioned so that when the cover is rotated the electrode traverses the circle of crucibles. Two viewing windows and three rotating handles are also screwed into the cover. Rubber O-rings provide vacuum seals between the various furnace components and permit complete rotation of the cover which is held down by a brass retaining ring screwed to the top ring of the main body. The electrode assembly consists of a copper tube with a central water channel and an annular return. It can be raised or lowered by means of an adjusting screw, and a modified Wilson-seal provides sufficient clearance for the tungsten tip to revolve in a 1-inch circle, thus ensuring that the arc can play over all parts of the charge. Power is provided by a Lincoln welding generator, and most melting is effected within the 200–400 A range at 15–20 V. A high frequency spark unit is used to superimpose a high frequency pulse on the welding generator voltage until the d.c. arc is established. A Welch Model 1397B mechanical pump evacuates the furnace to 2 μ in less than 5 minutes. Melting is carried out in argon, helium or argon/helium mixtures at a pressure of one pound. When necessary the gases are purified by melting a zirconium getter button in one of the crucibles for about 1 minute before working on the remaining crucibles.

Letter by S. T. Zegler & J. Terandy *Metal Progr.* **70**, 1956 116, 118

Homogenous Alloy Ingots Produced by Consumable Electrode Arc Melting

142/III

United States. Various methods of introducing alloying materials into zirconium-alloy ingots have been tested to determine their influence on the homogeneity of the ingot. The effects of magnetic stirring and of double melting on ingot quality have also been studied. In consumable electrode arc melting homogeneity is likely to be affected by the fact that alloying additions can only be made to that part of the ingot that is molten at any one time and the process is conducive to segregation. In the experiments, arc-melting was carried out in a water-cooled copper crucible in an atmosphere of $\frac{1}{5}$ argon and $\frac{4}{5}$ helium at atmospheric pressure, using a continuous, 2-inch. square electrode of pressed sponge. As many as 30 or more ingots, each 6–10 inch. dia. and weighing 300–500 lb. were melted, using each method. The test results are considered representative of production practice. Additions of tin were made to Zircaloy I, an alloy containing 2.5% tin, by (*1*) the pressed-in and (*2*) the tin-chopper method. For the first method tin wire $\frac{1}{16}$ inch diameter was incorporated with the zirconium sponge before pressing the electrode. This technique might be suitable for alloying materials of higher melting points, but it was unsuccessful with tin since the tin melted and ran out of the rod some distance away from the molten end. As a result an excessive amount of tin appeared in the lower end of the ingot. During the latter part of casting the flow rate of tin was apparently proportional to the melting rate of the electrode and tests showed a uniform, though low, tin content throughout the upper and greater part

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of the ingot. For the second method, short lengths of $\frac{1}{4}$ inch tin wire were fed to the molten pool from a wire chopper housed in a vacuum-tight box. The chopper consisted of four rubber-covered drive rolls geared to a rotary cutter which was driven through a vacuum seal by the electric motor which also drove the electrode feed rolls. This method resulted in areas of very high tin content because pieces of tin or zirconium-tin compounds sank to the bottom of the molten pool and solidified before completely mixing with the zirconium. Magnetic stirring was found to overcome this difficulty. Trials showed that with a coil of 4,000 turns of magnet wire wound around a non-magnetic water jacket, a current of 0.2 A gave a field which mixed the molten pool enough to produce sound ingots. Unalloyed tin was not detected in macroetched internal sections from such ingots. The quality of the ingots was improved by double-melting to eliminate porosity. The second melting was at first carried out in the same furnace and it was necessary to press-forge and roll the ingots to obtain electrodes of the required 2 × 2 inch. section. A disadvantage of this practice was that undissolved tin beads in the ingot gave a centre weakness that often resulted in failure during rolling. Trouble was also caused by oxide and nitrogen pick-up. Better quality ingots were obtained when a larger furnace was constructed to remelt electrodes composed of several 6-inch. diameter ingots connected end to end. Ingots produced by double melting in either furnace, however, were free of porosity and segregation. In the case of a quaternary alloy, Ziecaloy 2, containing 1.5% tin, 0.12% iron, 0.10% chromium and 0.05% nickel, the following addition methods were tested: (*1*) adding iron and chromium in the briquetting operation to sponge pre-alloyed with nickel, (*2*) alloying the nickel with tin wire, and (*3*) using tin-iron-chromium-nickel alloy wire extruded from blended powders. Although these methods gave fair-quality ingots they were less successful than a method of feeding compressed pills made from powdered metal into the melt during casting. Pills $\frac{3}{8}$ inch diameter by $\frac{1}{4}$ inch thick were compacted in a high-speed commercial tablet machine from blended tin, iron, chromium and nickel powders. A pill counter geared to the electrode feed motor gave additions at a rate proportional to the electrode melting rate. The use of the pill method resulted in better homogeneity than that obtained by any other method tested, and, in conjunction with magnetic stirring and double melting, yielded alloy ingots meeting rigid specifications. Reproducibility within the tolerances set for each alloy constituent was easily maintained.

Paper by R. A. Beall, F. Caputo & E. T. Hayes *U.S. Bureau of Mines Report* 5200 March 1956

143/III

Vacuum Melted High Temperature Alloys

United Kingdom. By about 1950 the temperature ratings of conventionally melted nickel- and cobalt-base alloys used for jet engine buckets had reached a limit set by the inability to increase the hardener content of the alloys without impairing the ductility. The development of better wrought alloys was also seriously limited by forging problems. The introduction of vacuum melting has removed these limits by giving higher ductility together with increased forgeability. It is not known how vacuum melting increases high temperature strength, but it is possible that the lower gas contents (up to 90% reduction compared to air melting), together with fewer oxide and nitride inclusions, play an important part in the mechanism of high-temperature strengthening. Vacuum arc melting has a further advantage in that the highly directional solidification—bottom to top— results in a greatly improved ingot structure. The alloys have also been improved by chemical modifications which have been made possible by vacuum melting: for example by the elimination of manganese and silicon, and by the increase in total titanium plus aluminium content made possible by the increase in ductility at a given hardener level. Other chemical modifications have been made as a result of investigations which showed that the inconsistencies observed in early vacuum melts were due to differences in composition not noted in casual chemical analyses of the melts. Research by the Universal Cyclops Steel Corporation has shown that very small residuals of boron (0.005–0.01%) and zirconium (0.05–0.1%) have a tremendous beneficial effect on the rupture life and ductility of various super alloys. These elements can be introduced accidentally by contamination. Zirconium may be introduced from a zirconia crucible. Boron contents of 0.002% may be introduced into vacuum induction melted heats by contamination from magnesia crucibles, and even this level of boron has been found to greatly increase the rupture life and ductility of the nickel-base, super-alloy Udimet 500. If correctly utilised a potent interaction between boron and zirconium can quadruple the rupture life and ductility of vacuum-melted, nickel-base super-alloys. Experiments in which the boron content was varied intentionally from 0.005 to 0.01% and the zirconium from 0.05 to 0.1% have indicated that variations within these ranges have no significant effect on rupture life and ductility. These are the maximum variations that should be encountered in vacuum melting the type of alloy considered with controlled boron and zirconium additions. Extensive tests have been carried out by Universal Cyclops on the mechanical properties of five nickel-base super alloys over the temperature range 1,000–1,600°F. In the case of three of the alloys, M–252, Waspaloy and GMR–235, vacuum melting plus the pertinent composition modifications resulted in a significant improvement in rupture properties over the range investigated. It seems possible that forged nickel- or cobalt-base alloys can be developed which will have a 100-hr. rupture life of 20,000 lb./inch^2 at temperatures approaching 980°C. This may represent the ultimate attainable in these systems and further increases in service temperatures of jet engines will require the use of refractory metals, such as molybdenum, for the critical bucket application. Nickel- and cobalt-base alloys will then be required, however, as turbine wheel materials. They may also be used in ram jets and rockets.

Article by Anon. *Metal Industry* 24.1.58 63-66

144/III

Manganese Losses in Melting Nickel-Manganese Alloys under Reduced Pressures

Germany. The Mn loss of alloys with Mn 0–10% was investigated in the temperature range 1,500–1,600°C and at 5 × 10^{-4}–5 × 10^{-1} mm. Hg in a high-vacuum induction furnace fitted with a rotary pump set S150/S5

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and an oil diffusion pump OT 1500. The logarithmic Mn loss was shown to follow the law

$$P = P_A \times 10^{-Kt}$$

where P is the Mn content (%), P_A the initial Mn content, t the duration of the experiment, and K a constant equal to $\log (P_A/P_E)/(t_E - t_A)$, P_E being the final Mn content at time $(t_E - t_A)$. This evaporation constant varied between 0.35 and 0.29 over a Mn range of 0.75–7.8% and between 0.32 and 0.68 over the temperature range 1,500–1,600°C, while the variation due to changes in pressure between 5×10^{-4} and 5×10^{-1} mm. Hg caused a change in K from 0.40 to 0.06. The loss was shown to be entirely due to evaporation.

Article by L. Schumann-Horn A. Mager & W. Deisinger &. *Metallk.* 47, March 1956 145-148

Molybdenum Alloys: When to Use Them

See Abstract No.: 219/I

145/III

Vacuum Furnace for Treating Titanium

See Abstract No.: 237/I

146/III

Arc and Vacuum Melting of Titanium I

147/III

United States. The chief features of inert electrode- and consumable electrode-furnaces are described and factors which influence the behaviour of the electric arc are discussed. Where inert electrodes are used, the metal is melted in argon and/or helium mixtures at approximately atmospheric pressure because the inert electrode arc goes over to glow discharge on melting in partial vacuum. The inert electrode furnace is used widely to produce small titanium ingots, or buttons, for determining sponge hardness range. Melting on a commercial scale is mostly carried out in consumable electrode furnaces. In these the electrode is made of titanium, either by a compacting process or by a preliminary melting operation. The feed of electrode into the furnace is usually governed by a servomechanism operated by voltage or current signals which depend on the length of the arc. Melting is preferably carried out in a partial vacuum. This has the advantage of removing hydrogen and other unwanted gases and volatile impurities. In addition, a more stable arc is obtained and the critical pressure at which glow discharge occurs is lower than in argon. Tests by the Bureau of Mines showed a critical pressure of 20,000 μ with a $\frac{5}{8}$-inch dia. welding electrode in argon, and a critical pressure of below 100 μ with a 4-inch. electrode in vacuum. Similar figures have been obtained in tests in which $7\frac{1}{2}$-inch' dia. electrodes were melted in the same furnace: the critical pressure was 10,000 μ in argon and 50–100 μ in air. It is not known how the arc is supported in a partial vacuum—whether by metal vapour from the anode or cathode or by the evolved gas. A heavy current has a stabilising effect on the arc at both low and atmospheric pressures. Lack of stability has sometimes been observed when the power is reduced at the end of a melt, although the use of low voltages may eliminate this. The voltage affects the shape of the electrode tip which is cone-shaped for a high-voltage arc and flat or mushroomed for a low-voltage arc. The depth of the molten pool is influenced by the type of arc, the melting pressure and the power input. For a straight polarity d.c. arc the depth increases as the pressure is decreased from atmospheric to just above the critical pressure. It is greater with a straight polarity arc than with either a reversed polarity or an a.c. arc. Some data on melting efficiencies are quoted in a table. Movement of the arc can be controlled from outside the furnace by permanent magnets or electro-magnetic fields and the arc may also be rotated by the field of a simple solenoid wound on the water jacket if the latter is of non-magnetic material. The article is based on work carried out by the Titanium Metals Corp. of America.

Article by D. E. Cooper & S. A. Herres *Metal Industr.* 7.12.1956 471-474

Arc and Vacuum Melting of Titanium II

148/III

United States. A solenoid-type magnetic field, such as that used to rotate the arc, sets up a constant agitation of the melt in a titanium arc furnace. This effect can be used to control the surface porosity, homogeneity and as-cast structure of the ingot. As the rate of stirring is increased the rate at which heat is removed from the melt is also increased and the size of the molten pool is therefore reduced. With moderate rates of stirring an improvement is obtained in surface porosity. At high rates of stirring the surface deteriorates. In tests by the U.S. Bureau of Mines too rapid stirring caused centrifuging of tin additions which were incompletely dissolved. The authors have shown that lack of homogeneity may be caused by failure to dissolve certain alloy additions when low melting currents are used and by freezing segregation. The latter can be minimised by induction stirring. Volatilisation of elements such as manganese during vacuum melting may give low alloy contents. Analyses made on 10 samples taken from a Ti-6% Al–4% V alloy melted at 8,000 A with induction stirring gave values of 5.71–5.83% Al and an even closer Vanadium range. Induction stirring has a significant effect on the as-cast grain structure since it breaks up the large columnar grains which are usually associated with ingot solidification. This effect is strongest when rapid stirring and hence a small molten pool are used. Its usefulness however must be considered in relation to the poor ingot surface resulting from rapid stirring.

Article by D. N. Cooper & S. A. Herres *Metal Industr.* 89, 14.12.1956 499-500

Production of Titanium Shaped Castings

149/III

United States. Methods in use for casting titanium parts have been investigated with the aim of determining the most suitable process for commercial application. The investigation has covered melting furnaces, mould materials and the development of casting alloys. Skull furnaces using a consumable electrode are considered

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the best for casting titanium. A furnace of this type with lip pouring has been used by the Bureau of Mines to cast titanium breech blocks weighing up to 75 lbs. Melting is effected rapidly under reduced pressure using high current densities of 8,000–9,000 A at 28–30 V with an 8-inch dia. electrode. Experimental work has shown that maximum melting rates are attained when the pressure of the furnace atmosphere is reduced to just above the value which would give rise to a diffuse arc. In the Bureau of Mines furnace the optimum operating pressure is 150 μ. A crucible 9–10 inch. in diameter is used for melting and casting 90–100 lb. of titanium and melts are prepared in about 15 mins. The melts have a high degree of superheat and sound castings can be obtained from graphite moulds without pre-heating. Sponge, ingot or scrap can be used for the electrode and most of the furnace operations can be mechanised. Arc furnaces with fixed electrodes have been used in research by Rem-Cru Titanium, Frankford Arsenal and Battelle. A commercial fixed-electrode, skull-melting furnace for titanium is made by the National Research Corporation. In this all melting is effected in an enclosed chamber under an inert atmosphere at a reduced pressure of about 200 mm. Hg. Castings weighing up to 50 lb. can be produced. Disadvantages of fixed-electrode furnaces are the difficulty of obtaining adequate superheat in the melts and the danger of contaminating the melts with tungsten or graphite from the electrode. Titanium can be melted and cast in graphite-lined induction furnaces but the castings generally have poor ductility because of carbon contamination. Other refractories such as zirconium and thoria also cause severe contamination. A new technique has been developed by Watertown Arsenal in which a strong-intensity electrical field is used to melt the titanium as rapidly as possible, and to force the melt to assume a very marked conical shape. The time and area of contact between the melt and the graphite crucible are thus reduced to a minimum, and 5 lb. ingots with carbon contents of 0.066 to 0.15% have been obtained from sponge containing 0.025% carbon. If similar results can be obtained in the furnace with its capacity increased to 25 lb. the method should be suitable for small castings in which a little carbon contamination can be tolerated. No suitable direct method of measuring the pouring temperature of titanium melts has yet been developed. An indirect method used at Armour and Battelle is to measure the power consumed after the titanium has been completely melted and to take this as a measure of the amount of superheat in the metal. Investigations into mould materials have shown that machined graphite is still the best material for producing smooth, sound castings with negligible surface contamination. Other mould materials contaminate the castings to a greater depth than machined graphite, but small castings can be made in expendable moulds composed of refractory oxides or graphite powder. An investment type of mould material is made by the National Research Corp. Little work has been done on the casting of titanium alloys but there is some indication that the alpha and alpha-beta alloys have good casting properties whereas the meta-stable beta alloys are difficult to cast. The largest potential market for titanium castings lies in the chemical process industry and, when costs are reduced, in marine and ordnance applications. In the U.S. aircraft industry there is a potential demand for 125 tons of titanium castings per year.

Report by R. M. Lang G. H. Schippereit & J. G. Kura *Titanium Metall. Lab. Rep. No.* 60 12.12.1956 34 pp.

150/III R. A. Stauffer & National Research Corp. *U.S. Pat.* 2,768,074

Method of Producing Metals by Decomposition of Halides

United States. A process for the continuous quantity production of zirconium, titanium, vanadium, chromium and hafnium, with a high degree of purity is described.

151/III

Problems of Gases in Metals

See Abstract No.: 47/I

152/III

Process for the Degassification of Molten Steels

Germany. In the method according to the invention steel which has been melted in atmosphere and is in a condition ready for casting, but at a higher temperature than normal casting temperature, is allowed to fall freely through a vacuum chamber. The rate of feed of the stream is controlled by using a variable diameter (25–70 mm.) inlet into the vacuum chamber and maintaining the liquid column of the stream above the inlet at a minimum height of 300 mm. The pressure in the chamber may vary from 30 to 0.1 mm. Hg with the result that the stream divides up into individual droplets of 10–10^{-3} mm. diameter, allowing maximum dissipation of gas. With correct choice of inlet size and chamber pressure, according to the type of steel being dealt with, the droplet size can be adjusted to effect degassification in a shorter period of time than has been achieved by previous methods.

Bochumer Verein Fü Gusstahl-Fabrikation A. G. *Brit. Pat.* 792,160

153/III

Transformer Steel

See Abstract No.: 229/I

154/III

The Industrial Treatment of Steel-Melts Under Reduced Pressure

Germany. A method suitable for degassing steel melts in vacuum on an economic industrial scale has been developed and plants are in operation in the Siemens-Martin and the Thomas steel works. The principles and construction of the plant are described. The time necessary for vacuum treatment is determined by the time taken for the carbon-oxide reaction. Removal of the carbon-oxide sets up a flushing effect which in turn

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Abstract No. and References

sweeps away hydrogen and nitrogen. Tests with non-alloy steel have shown that the equilibrium of the carbon-oxide reaction is reached in less than 1 minute at 10 mm. Hg. Dross in the melt should be removed or rendered inert to prevent it reacting with the melt to form oxygen. Reaction with refractory linings is not of great importance because of the small area of surface contact and the short treatment time. In practice the life of the lining is prolonged by using strong brickwork and suitable forms and by avoiding unnecessary temperature stresses. Degassing is carried out between the tapping of the blast furnace and pouring, so manufacturing and casting methods can remain unaltered. In the test plant the vacuum chamber is mounted on a platform which can be moved by a crane. The chamber has a height of about 2 metres to allow for the strong frothing which accompanies degassing. A suction pipe connected to the bottom of the chamber dips into a pouring ladle containing 40–80 tons steel. By lowering and raising the chamber through a distance of about 60 cm. a charge of about 4 tons is alternately drawn into the chamber for degassing and then returned to the ladle. This process is repeated until the desired degree of degassing has been obtained throughout the metal in the ladle. The number of dips necessary can be determined mathematically. Three rotary piston pumps and a Roots-blower evacuate the system and gas coolers fitted with dust separators are used. The process can be controlled by observing the pressure changes and the composition of the gas drawn off. Attainment of the minimum carbon and oxygen content can be checked by the pressure changes which occur when oxygen or carbon carriers are added through a vacuum lock. With this plant a ladle of 80–100 tons of Siemens-Martin steel is processed in 30 minutes by degassing in steps of 4 tons for 30 secs. The oxygen and hydrogen contents are reduced to about a third of the initial value. With low oxygen contents the harmful effects of nitrogen can be avoided by adding small quantities of nitride formers. The forming, forging, segregation and etching properties of the metal are found to be better than those of untreated steel. It should prove more resistant to corrosion and is particularly suitable for galvanising and for surface treatment by metal diffusion, *e.g.* chromium diffusion. An advantage in manufacturing is the speed at which high quality can be obtained. The metal can be re-melted in the arc furnace without need for degassing and a refining period can be omitted from the arc furnace steel process if the sulphur content permits. Extra charges and alloy additions do not require a preliminary heating to prevent hydrogen pick up by the steel. In the plant installed in the Thomas steel works the ladle and not the degassing chamber is moved. Pumping is effected by a Roots-blower and a water-ring pump at a speed of 15,000 m.³/h. Dust particles with a diameter of more than 5 μ are separated out in a cyclon. The dome of the chamber is made of carbon bricks and an indirect arc heater of 3,000 kW makes it possible to heat a 30-ton melt through about 30° in 10 minutes during treatment. With longer treatment times this heating permits the addition and melting of alloy elements under vacuum. About 30 tons of Thomas-steel melt are treated in 10 minutes. The Siemens-Martin plant resembles the test plant with a suction speed of 6,000 m.³/h. and a heating power of 400 kW. About 80 tons of steel are treated in 30 mins.

Article by F. Harders H. Knuppel & K. Brotzmann *Stahl u. Eisen* **76**, 27.12.1956 1721-1728

Vol. 7-8 57/58

Apparatus for Extracting Gases from Molten Metal

155/III

Germany. The patent describes a device for arresting condensates of gases extracted from molten metal on subjecting the melt to a reduced pressure. A filter element is contained in a filter housing through which passes a stream of gases from the melting vessel to a suction pump. Filtration is achieved by a plurality of long cylindrical cloth sacks closed at their bottom ends and opening at their upper ends into the housing space near the outlet duct. The sacks are kept in shape by inner rings and by being attached at their lower ends to a relatively heavy plate. The stream of gas passes into the filter unit and is deflected by a protective baffle so as to pass through the filtering elements in an upward direction, and thence to the pump. Filter cloth in which a specimen area of 20 cm.² permits the passage of 150–180 l. of air in 5 min. at 20 mm. water pressure difference has been found suitable. The preferred subatmospheric pressure for the process is 20 mm. Hg although pressures between 30 mm. and 0.1 mm. Hg may be used. The working pressure is controlled by valves on the inlet and the pump sides of the system, and the latter may be used to clean the filter elements by a 'blow-back'.

Bochumer Verein Für Gusstahl-Fabrikation A. G. *Brit. Pat.* 782,558

Practical Processes for the Degassing of Light Alloys

156/III

See Abstract No.: 48/I

Vacuum Treating Non-ferrous Melts

157/III

(Reference only)

Article by V. DePierre & S. Inouye *Foundry* Dec. 1956 108-114

A Vacuum Fusion Gas Analysis Apparatus for Use in the Quality Control of Ferrous Materials

158/III

United Kingdom. It is useful to be able to make a gas analysis (especially for O_2, N_2 and H_2) of a melt of steel before tapping the bath; in vacuum melting, using gaseous H_2 to reduce impurities, this would be particularly useful. The article describes an apparatus developed for the purpose which consists almost entirely of metal and avoids the use of greased joints. The samples are melted by induction in a graphite crucible contained in a fused silica tube, 17 inch. long and 2½ inch. in diameter, which is surrounded by a Perspex water jacket. The tube is sealed by a Neoprene gasket to a 3 inch. steel header which connects *via* two right-angle bends to

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a 4-stage mercury diffusion pump, backed by a rotary pump. A glass connection is made to a McLeod gauge from the top of the header, which is also fitted with a quartz window through which a pyrometer can be sighted on the sample, and with an air-lock. After pumping to about 10^{-4} mm. Hg, with no sample in the crucible, the induction heater is switched on and the temperature raised to 2,400°C which is maintained until the pressure falls to 5×10^{-5} mm. Hg. It is then reduced to 1,600–1,650°C and the blank rate determined. This should be not more than 0.02 ml. per hour. The gas evolved is analysed by valving off the rotary pump and drawing the gas with a Toepler pump through a trap containing CuO at 600°C, a P_2O_5 trap and a freezing trap. A small three-stage mercury diffusion pump then returns the remaining gas to the calibrated bulbs, the pressure being measured after each component is removed, only O_2 and N_2 remaining finally. The vacuum lock for charging the furnace was not altogether successful and the samples were moved from a side arm by a magnet and lowered into the furnace. The equipment takes up a floor space of only 3¼ ft.² and is readily movable.

Article by K. Speight & G. M. Gill *Metallurgia* March 1957 155-158

159/III

A Study of the Hydrogen-Pressure Relationships in the Zirconium-Hydrogen System

See Abstract No.: 250/I

160/III

Vacuum Fusion Determination of Oxygen and Nitrogen in Lanthanum

United States. Using nickel as a bath for the samples, reliable analyses of oxygen in lanthanum have been made by the vacuum fusion technique. The method is also suitable for determining the nitrogen and hydrogen contents. A nickel bath was chosen since fusion of lanthanum in a graphite crucible at temperatures up to 2,000°C did not yield a quantitative recovery of oxygen or nitrogen, and with an iron bath, evolution of carbon monoxide and nitrogen was incomplete at temperatures up to 1,850°C. The furnace used consisted of a 90-mm. borosilicate glass cylinder with a ground-glass seal at the bottom. A Vycor crucible containing an Acheson graphite heating crucible was supported in the cap closing the cylinder. Gases from the furnace were pumped into the analytical section by a three-stage mercury diffusion pump. The analytical unit consisted essentially of two known volumes and an accurately calibrated McLeod gauge. At the top of the McLeod gauge capillary tube was a three-way high-vacuum stopcock with a standard taper joint for the attachment of gas sample bulbs. An oil diffusion pump evacuated the gas bulbs through the third stopcock position and also evacuated the remainder of the analytical section. The mercury for the McLeod gauge and the cutoffs was raised by atmospheric pressure and lowered by means of a small auxiliary mechanical pump. The collection system and McLeod gauge could collect up to 4×10^{-4} mole of gas, and the amount of gas could be measured to a precision of about 0.3% at 1×10^{-4} mole and about 1.6% at 6×10^{-6} mole. When a gas sample had been collected and its pressure measured, part of it was forced into the sample bulb using the McLeod gauge as a Toepler pump. The relative proportions of the gases present were determined with a mass spectrometer by an isotope-ratio method. Lanthanum for standard specimens was prepared by calcium reduction of lanthanum fluoride followed by melting in magnesium oxide at 1,050°C under vacuum. Pieces of this metal were melted at 1,200°C under vacuum to form small cylinders. Some samples cut from these cylinders were used as blanks and others were heated in known quantities of oxygen to form oxygen standards for checking the accuracy of the method. To prevent atmospheric contamination lanthanum samples were cleaned and weighed in an argon-filled glove box. Whilst still in the box they were placed, together with the nickel, in a loading tree which was then transferred to the vacuum fusion apparatus. The apparatus was evacuated and the temperature of the crucible was raised to 2,300–2,400°C for about three hours to degas the furnace. By this time the pressure in the furnace had usually fallen to about 5×10^{-5} mm., as measured with a cold cathode gauge. The crucible temperature was lowered to 1,300–1,400°C and the nickel was dropped in. Then the temperature was raised to 1,900°C and a blank run was made after which the temperature was lowered to 1,300–1,400°C before adding the lanthanum sample. The temperature was again raised to 1,900°C and evolved gas was collected for 15 minutes. It was found possible to recover, as carbon monoxide, an average of 90.9% of the oxygen added to the standard samples. Incomplete recovery of oxygen is probably due to adsorption of the evolved gases by nickel evaporated on the upper walls of the furnace tube and analyses must be corrected to allow for this. Using a nickel bath at 1,600°C complete recovery of nitrogen was achieved. As hydrogen is easily evolved and should not be adsorbed to a greater extent than carbon monoxide, the method should give accurate results for hydrogen.

Article by D. T. Peterson & D. J. Beerntsen *Analyt. Chem.* **29**, Feb. 1957 254-257

161/III

Determination of Oxygen in Niobium

See Abstract No.: 210/I

162/III

Zone Melting

See Abstract No.: 53/I

163/III

Preparation of Uranium Metal

See Abstract No.: 215/I

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Zone Melting of Uranium

164/III

United Kingdom. It is shown that the process of zone refining can be applied to uranium, but that metallic impurities require slow zone speeds ($\frac{1}{4}$ inch/hr.). Non-metallic impurities can be removed using higher speeds (3 inch./hr.). The purification processes are more complex than in simple zone refining. If the bar of uranium is held vertically and the zone moved upwards, the high density of uranium helps separation. Compounds between metallic and non-metallic impurities are also formed, so that under certain circumstances the addition of, for example, oxygen helps the removal of metallic impurities. The method is useful for the direct purification of uranium and for removal of fission products. The experiments were carried out using a vacuum molybdenum-tube resistance furnace. The tube was 1 inch in diameter, 2 inch. long and 0.005 inch thick. Beryllia crucibles were used, and the zone, $1\frac{1}{4}$ inch. long, was at a temperature of the orderof 1,200°C. The vacuum was obtained using Edwards oil diffusion and backing pumps, and was better, than 0.01 micron.

Report by J. E. Antill E. Barnes & M. Gardner *A.E.R.E. Rep. No.* M/R1985 June 1956

The Floating-Zone Melting of Refractory Metals by Electron Bombardment

165/III

United Kingdom. The authors have used an electron bombardment method of heating the vertical rod used in this technique employing an annular tungsten cathode with a high voltage applied between it and the specimen rod. This has the advantage of a concentrated heat output enabling refractory metals to be zone-melted. The process is carried out in vacuum allowing three processes to contribute to the ultimate purity of the specimen (1) outgassing, (2) evaporation of volatile impurities, and (3) zone-refining. The authors discuss the size limitations imposed by the method and also present a limited mathematically treatment of the temperature distribution along the rod.

Article by A. Calverley M. Davis & R. F. Lever *J. Sci. Instrum.* **34**, April 1957 142-147

Refining Zone Refining

166/III

United States. Describes an automatic method of purifying (*e.g.* silicon by 'zone refining'). A rod of the material is held vertically in chucks inside a silica tube with inert gas atmosphere and heated over a short length only by r.f. (10 kW, 4 Mc./s. generator). The rod starts with its lower end level with the r.f. coupling coil and is heated to about 800°C by passing a current along it. At this temperature, it absorbs about enough r.f. power to heat it to 1,450°C and a short section (or zone) near the end melts. The equipment then automatically moves the rod downwards, rotating it at the same time, so that a molten zone effectively travels up the rod. Impurities dissolve preferentially in the molten zone. When the zone reaches the upper end, the power is reduced so that the zone solidfies and the rod is lifted up ready to start again. The process can be repeated automatically as often as desired; results quoted say that 67 cycles gave p-type silicon with a resistivity of 16,000 ohm. cm. indicating impurity levels of less than one part per billion. The note also refers to an improved method of bonding soft metal conductors (*e.g.* Au, Ag, Al) to semiconductor surfaces by 'thermo-compression bonding', a combination of high pressures and temperatures well below the m.p.

Article by Anon. *Chem. & Engng. News* **35**, 12.8.1957 88–90

The Development of the Iodide Process of Refining Titanium and Zirconium

(Reference only)

167/III Article by R. A. J. Shelton *Metallurgia* May 1957 225-231

Process of Smelting Germanium

168/III

Germany. Improvements in the quality of substances such as high-purity germanium and silicon, and more rapid and effective smelting are claimed to result if the quartz smelting crucible is coated with a thin layer of amorphous carbon. Such a layer gives off only little gas in high vacuum, and does not dissolve in germanium or the like. The molten product can be easily dropped out of the crucible without damage to the latter, whereas uncoated quartz crucibles must be broken to remove germanium attached to the walls. Coating with carbon is effected as follows. The crucible is placed in a tube which is oven-heated to a temperature of about 300°C. A container of benzene or other hydrocarbon is then connected to one end of the tube and a water jet or other suitable pump is connected to the other end *via* a valved conduit and a trap maintained at room temperature. After pumping has been started the benzene is placed in a temperature bath of 0°C, then for 10–12 minutes the temperature of the tube is increased to 830°C while the pump operates continuously. The pump is then turned off and the tube temperature is raised to 900°C for 20 minutes. The hydrocarbon is cracked at this temperature, which is below the softening point of quartz, and a thin film of amorphous carbon is deposited on the crucible. The oven is then allowed to cool to 300°C or 200°C and the coated crucible is removed from the tube. From time to time the amorphous carbon coating should be burned off and the quartz crucible recoated.

K. O. Seiler & International Standard Electric Corp. *U.S. Pat.* 2,780,539

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38 — DISTILLATION — 38

169/III

Short-Path Fractionation Apparatus

Liechtenstein. A short-path fractionation apparatus which simultaneously distils separate molecular fractions, is described. The mixture to be distilled is spread as a film over the heated troughlike surface of the distillation flask. This surface has a temperature gradient which causes simultaneous distillation of different fractions, to be collected in separate cooled receivers. As in a previous apparatus (*Chemical Abstracts*, **45**, 7392d), the pressure corresponding to the boiling point is maintained by capillary tubes which connect the distillation flask with the reservoir.

(*Chemical Abstracts*)

Gerätebau-Anstalt
Swiss Pat.
305,408

170/III

Testing of a Rotary Concentric-Tube Distilling Column

United States. In 1947 a description of the construction and performance of a small (3 inch) rotary concentric-tube distilling column was published. Its efficiency was ten times that of any other previously reported rectifying column. Extrapolation of the performance figures led to the design of the large column described here. The rectifying section of the column consists of an annular formed between two vertically concentric stainless steel cylinders. The annular is 0.0465 inch in width, five feet long and has an inner diameter of 4.871 inch. The inner cylinder can be rotated at speeds between 1,000 and 8,000 r.p.m.; the outer one is surrounded by a four-element heating jacket and insulating material. Built-in thermocouples are used to monitor the cylinder wall and jacket temperatures at uniformly spaced points along the tube. The column is mounted on an electrically heated 3-gallon stainless steel pot. The drive to the inner cylinder and the condenser are located at the top of the column where the temperature of the vapour liquid equilibrium is measured with a thermocouple having ten junctions. 2, 2, 4- trimethylpentane was used to test the transmission of the column, methylcyclohexane being added to test separation efficiency. The variables were cylinder speed and heating power input to the pot which was altered in 50-watt steps from 200 to 400 watts. The jacket heating power was generally chosen so that the wall of the outer cylinder and the jacket were at the same temperature. The column transmission is directly proportional to the power input to the pot and to the power into the heating jacket. Associated with increased transmission is a reduction in separating efficiency. Test data show that the separating efficiency of the 5-inch. column at speeds of rotation above 2,400 r.p.m. is much lower than could be expected from a linear extrapolation of the data of the 3-inch. column to high speeds of rotation. This fall-off in performance is due to superheating above the equilibrium temperature of the vapour in the annular space by heat generated through friction in the vapour.

Article by
B. J. Mair
N. C. Krouskop &
F. D. Rossini
Analyt. Chem.
29, July 1957
1065-1068

171/III

Centrifugal Boiler Compression Still

United States. A compression still is described which has a distilling capacity four to seven times that of the present stationary type of equivalent size.

Article by
K. C. D. Hickman
Indust. Engng. Chem.
49, May 1957
786-800

172/III

A Preliminary Study of Some Aspects of a Heavy Water Plant

United States. Most of the critical cryogenic problems surrounding the construction of a large heavy water plant based on the fractional distillation of liquid hydrogen and hydrogen deuteride have to a large extent been solved during the past few years. Efficient methods of handling liquid hydrogen in large quantities have been developed. Very efficient types of insulation are available and refrigeration at the 20°K temperature level can be obtained in a reliable and economical manner. The major problems of fractional distillation of liquid hydrogen and its isotopes have shifted to column design. At the present time no engineering data for large-scale distillation are available. Without them minimum dimensions for a column cannot be calculated and consequently factors such as cool-down time, time required to obtain equilibrium, and amount of refrigeration required cannot be accurately determined. However, even with the few distillation data in existence it appears that fractional distillation of hydrogen isotopes provides a very efficient process for the production of heavy water or deuterium gas of high purity. It is felt that purification of the feed gas is a separate problem which should be considered in connection with potential supplies of raw feed gas. There are probably a number of potential sites for a heavy water plant in the Unites States, each one of which might require a purification system somewhat different from the others. Finally, the estimates made in this report indicate that electrical energy consumption per pound of heavy water will be of the order of 630 kWh. The product obtained from the column consists of 3% hydrogen deuteride and 97% hydrogen. Little additional energy is required to concentrate this product to essentially pure deuterium or heavy water.

(*Nuclear Science Abstracts*)

Report by
P. C. V. Arend
N.B.S.
*Rep. No.*NBS.3579
February 1955

173/III

Composition of Lubricating Oil

United States. Information on the molecular structure of hydrocarbon types has been obtained by a study of the composition and properties of fractions separated from a lubricating oil. The separation was effected by

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a process of molecular distillation, silica gel chromatography and liquid thermal diffusion. Starting with 5.5 litres of oil having a molecular weight range of C_{19} to C_{50}, a series of distillations was made in a 20-stage molecular still at a pressure of 1 μ. Fourteen separate equilibrium-type distillation runs and one batch distillation run were necessary to process the oil. The boiling range of the resulting fractions was 196° to 535°F (at 1 μ). The middle cut having a boiling range of 370° to 431°F and a volume of 1.5 litres was re-charged to the still for a second distillation. In this run the still was operated as a batch distillation unit at a throughput of 40 ml. per hour and a reflux ratio of 20 : 1. A heart cut having a boiling range of 393–414°F, a molecular weight range of C_{31} to C_{39} and a volume of 565 ml. was selected and aromatics were removed by elution chromatography. The narrow boiling saturated hydrocarbons were then separated according to molecular type by thermal diffusion in a battery of 10 columns. At the end of 10 days 10 fractions were collected. A further separation was carried out on each of these fractions and a total of 100 fractions was obtained. A third continuous run was made by arranging these fractions in groups of increasing refractive index and charging the groups in turn to a single 8-ft. thermal diffusion column. A total of 43 fractions of about 5 ml. each was obtained from the run. These fractions were finally purified by percolating over alumina in micro-adsorption columns. The average number of rings per molecule was calculated for each fraction according to the n–d–M ring analysis method of van Nes and van Westen, using refractive index and density measurements and the average molecular weights found by mass spectrometry. Numbers ranging from 1.7 to 7.2 through the fractions were obtained. An independent calculation from the carbon-hydrogen ratio gave a range from 0.5 in. fraction 2 to about 6 in. fraction 43. Hydrocarbon-type analyses were made by high temperature mass spectrometry and ultraviolet spectrometry. Infrared spectrometry was used to measure the total methyl and methylene groups present in aliphatic paraffin chains. Mass spectrometric analyses were made using the parent ion method of calculation and the fragment ion method. The average number of rings per molecule calculated from parent ion analysis data was found to lie midway between the two values previously calculated. The work indicates that two general groups of hydrocarbon types may be responsible for the characteristics of the oil. The first group includes fractions 1–34 and is composed largely of isoparaffins and cycloparaffins containing 1–6 rings or an average of about 2 rings per molecule. These fractions showed the favourable properties of the oil such as good viscosity, viscosity index and pour point. The second group includes fractions 35–43 and contains cycloparaffins having 3–10 rings or an average of about 5 rings per molecule. In this group the viscosity and pour point increased and the viscosity index decreased rapidly as the number of rings per molecule increased. As the number of rings increased the length of aliphatic chains decreased. Fraction 43 was found to have unusual properties. It was a colourless, thick, tacky liquid which did not flow visibly below a temperature of 115°F. It consisted mainly of multi-ring cycloparaffins containing at least four and as many as 10 rings per molecule. Appreciable concentrations of 8–, 9– and 10–ring hydrocarbons were present in this fraction.

Paper by
F. W. Melpoder
R. A. Brown
T. A. Washall
W. Doherty &
C. E. Headington
Analyt. Chem.
28, Dec. 1956
1936-1945

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The Nature of Vanadium in Petroleum

United States. A centrifugal molecular still, Model CMS–5, was used for distilling vanadium compounds from various extracts. Excess castor oil was used as carrier oil, a reference oil, and a diluent to minimise cracking and coking.

174/III
Article by
L. K. Beach &
J. E. Shewmaker
Ind. Engng. Chem.
49, 1957
1157-1164

Drying Viable Biological Materials by Solvent Extraction and Azeotropic Distillation

United States. The biological material is mixed with n-butyl acetate and azeotropic distillation carried out in a steam-jacketted agitated pot still under an absolute pressure of 135 mm. Hg.

175/III
Article by
R. R. Freeman
M. A. Auro
T. R. Daskiell
J. E. Murphy
I. Oshrine &
R. F. Smith
Chem. Engng. Prog.
53, 1957
591-592

Flash Evaporator for Continuous Distillation of Sea Water

United Kingdom. A new design of flash evaporator in which the whole of the equipment can be accommodated in a single vessel is described.

176/III
Note by
Anon.
Engineer
204, 18.10.1957
572

Removing Milk Off-Flavours

See Abstract No.: 185/III

177/III

Abstract No. and References

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39 — MISCELLANEOUS PROCESSES — 39

178/III

Article by J. N. W. Payne *Petroleum* June 1957 221-224

Aluminium and Lithium Greases

United Kingdom. This article describes plants used for the production of aluminium and lithium greases by continuous methods. It is claimed that these enable a better product to be obtained economically with less hazards than are present in the case of batch processes. Typical systems are described for the production of both aluminium greases and lithium greases and details are given of the 'Votator' heater and cooler units used.

179/III

Note by Anon. *Packaging News* March 1957 13

Gas Filled 'Cushion' Keeps Cut Flowers Fresh

United Kingdom. Experiments have been conducted on the packaging of cut flowers for transport and preservation. Transparent envelopes of polythene-coated cellulose film were used, which were evacuated by a Vac-U-Seal machine, which required only the mouth of the bag to be inserted into the machine. After evacuation, the envelopes were filled with nitrogen or carbon dioxide, and then heat-sealed by the same machine. Shop-bought flowers were still in perfect condition ten days after sealing, though it is thought that the presence of moisture shortens the life of the flowers.

180/III

Simple Vacuum Tester for Canned Foods

See Abstract No.: 56/II

181/III

Article by M. L. Seaman *Food Engng.* 29, March 1957 90-91

Better Way to Measure Can Vacuum

United States. A new vacuum gauge is described which greatly minimises the uncertainty and errors which arise when the can is punctured due to the introduction of air into the can headspace. The gauge consists of a metal block from the base of which protrudes the hollow needle used to puncture the can; the mechanical vacuum gauge is mounted on top of the block. The hollow needle connects to a small space at the underside of a rubber diaphragm. A tube leads from the centre of the upper side of the diaphragm to an outlet which is connected to a vacuum line. An annular groove in the block containing the above tube is connected *via* a second tube to the vacuum gauge and also, *via* a bleed, to atmosphere. Before the can is punctured the diaphragm pressing against the block isolates the two tubes; thus the gauge is connected to atmosphere *via* the bleed. When the can is punctured, the diaphragm is drawn down and remains in this position until the gauge pressure falls to the can pressure. The diaphragm then moves up to seal off the gauge from the vacuum line and the gauge reads the can pressure. The bleed then causes the gauge pressure to rise and the diaphragm again falls and the above cycle is repeated. In practice, the gauge reading is substantially constant, while the diaphragm describes a rapid fluttering action. Data is given to compare the accuracy of this gauge with previous types.

182/III

Continuous Vacuum Dehydration Reduces Cost

See Abstract No.: 114/III

183/III

Storing Freeze-Dried Beef

See Abstract No.: 32/IV

184/III

Article by Anon. *Food Manufacture* 1.10.1957

The Use of Vacuum in the Manufacture of Spaghetti

United Kingdom. It has been found that the removal of air by exposure of the dough to vacuum improves both the appearance and mechanical properties of spaghetti. It is difficult to apply the vacuum to the mixing chamber because dust from the dry ingredients tends to be drawn into the pumping system and because it makes the introduction of the raw materials and the removal of the dough more complicated. A machine is described in which the vacuum is applied to a special chamber which connects the open mixing trough to the screw press used to extrude the spaghetti. The dough is conveyed to the chamber by a screw conveyor, the dough itself making a seal to prevent the access of air. The vacuum chamber is equipped with revolving blades, which break the dough into small pieces to facilitate the removal of air.

185/III

Removing Milk Off-Flavours

United States. Six types of vacuum equipment for removing off-flavours from milk by a fractional distillation operation are described. The Aero-Vac Flavorizer, made by the Cherry-Burrell Corp. uses two vacuum chambers, the first held at a vacuum of 9 inch. Hg and the second at 22–25 inch. Hg. Milk is sprayed into the first chamber countercurrent to a flow of steam which heats it just up to boiling point, thereby driving off some of the volatile off-flavours. The steam condensed in heating up the milk is removed, as the milk flashes

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into the second chamber, and carries away additional off-flavours. A similar two-chamber operation is used in the Vac-Heat process of the Creamery Package Mfg. Co. In the Para-Vac Process of APV Dairy Division Inc. a single vacuum chamber is used in which the milk is sprayed countercurrent to an upwards flow of steam. The vacuum and temperature are adjusted to prevent either concentration or dilution of the milk. In the Supervacuumizer developed by the Chester-Jensen Co. milk is passed into a vacuum tank operated under flashing conditions at about 25 inch. Hg vacuum. The walls and bottom of the chamber are cooled with a water jacket to recondense the vapours formed by flashing so that there is no measurable product loss. Two vacuum treatments with an intermediate heating stage are used in the Vacu-Therm process of the De Laval Separator Co. The milk is first heated to 138°F and is subjected to vacuum treatment in the first chamber. It is then heated to pasteurising temperature and vacuum-treated in a second chamber. The temperature and vacuum in the second chamber are adjusted so that the milk is flash vaporised. The vapours are introduced as 'steam' into the first chamber where they condense. No real steam is injected into the chamber. The sixth equipment, the Vacuum-Pressure Process Vat, manufactured by the Pfaudler Co., is designed for batch processing milk in quantities of 500, 800 and 1,000 gallons. The vat can be heated or cooled over its sides and bottom, and a sweep-type agitator maintains flow over the heat-transfer area. A single motor-driven pump provides the vacuum and also the pressure for discharging the milk.

Note by Anon. *Food Process.* Dec. 1956

This Unit Puts Freeze-Drying Right on the Processing Line

186/III

United Kingdom. Kestrel Products Limited, Aberdeen, are freeze drying fish, steaks, chops, fruits and vegetables on a commercial scale. Flavour retention is better than with conventional methods of preservation and long storage without refrigeration is possible for the products in any climate. Reconstitution in water is rapid. A charge of 700 lb. of material can be dehydrated in 6 hours. Fish, which are being supplied to West Africa, are desiccated to 5% moisture content. The drier is compact. A vacuum chamber 5 ft. long and 6 ft. in diameter with doors at either end holds 18 contact plates $2\frac{1}{2} \times 5$ ft. for the material. The temperature of the plates is controlled by passing hot water through them during drying and cold water for the final cooling of the product. A hydraulic mechanism applies pressure to each plate during drying. A vacuum of 0.21 p.s.i. absolute is drawn during drying by steam ejectors working into a jet condenser. The condenser and cooling water are centrifugally pumped from this and non-condensable gases are removed by water ejectors. Maximum steam consumption is 1,000 lb./hr. at 120 p.s.i. and 4,700 g.p.h. cooling water are used. A flow sheet for the production of meat bars, a seasoned blend of dehydrated beef and pork freeze-dried in this plant and vacuum-packed in foil-film laminated pouches, is given.

Article by Anon. *Food Engng.* **28**, Nov. 1956 40-41

Freeze Grinding

187/III

United Kingdom. A new technique of grinding at low temperatures using liquid nitrogen as a cooling fluid is described. It is suitable for materials, such as certain foodstuffs, pharmaceutical products and plastics, which are sensitive to heat or oxidation or which are volatile and cannot be suitably ground by other means. In some cases, as with polythene for flame spraying, the resulting particles have a size and shape more suitable for subsequent operations than those obtained by ordinary methods. The grinding is effected with the fixed or loose hammer-type of mill operating at high speeds. Stainless steel machines of the type used to grind foodstuffs are recommended since the construction material must withstand operating temperatures of below −40°C. In a plant constructed for Dohm Ltd. the particles to be ground are cooled in a heat-exchanger mounted above a high-speed pulveriser. Since ordinary lubricants might freeze, the bearing housings of the mill are lubricated with a molybdenised oil of low viscosity. The heat exchanger consists of a cylinder within a cylinder, the interspace being thermally insulated. To compensate for the contraction of air in the interspace on cooling an air inlet is provided through a small drying tower containing silica gel. An electrically-operated vibrator moves the material along. Liquid nitrogen is injected at the lowest point of the heat exchanger drenching the feed just prior to its transfer to the pulveriser. Compared with liquid oxygen and carbon dioxide refrigerants liquid nitrogen has the advantages of being chemically inert and of having a lower boiling point (−195.8°C). It makes intimate contact with an exposed surface and provides rapid chilling. Liquid nitrogen must be stored and transported in thermally insulated vessels fitted with a vent to the atmosphere. Evaporation losses are inevitable and the liquid cannot be stored indefinitely. A well-constructed storage vessel with an evacuated interspace filled with powder will not lose more than 3% per day when operated at the designed interspace pressure. The liquid may be delivered to stand or mobile tanks or small quantities may be delivered in 5 gal. Dewar vessels. When using liquid nitrogen it is advisable to wear asbestos gloves and to protect exposed glass over which the liquid may be poured. Experimental work on freeze grinding has been carried out using polythene and nylon. Polythene was difficult to grind even at low temperatures but particles of 40-mesh size could be obtained at a production rate of about 40 lb./hr. The estimated increase in cost due to the processing was about 80% of the raw material cost. Nylon was found to be more friable than polythene and could be processed for an increase of about 12% on the raw material cost. Advantages of the method which may lead to overall cost reductions are the high speed of pulverising and the small floor area required for the equipment. Its main disadvantages are the increased complexity of the grinding plant and the need to forecast liquid nitrogen requirements.

Article by A. Bracken & L. J. Brittaine *Mfg. Chem.* **27**, Dec. 1956 497-499

Vacuum-Insulated Transfer Tube

188/III

United States. The system is designed for use in the transfer of liquefied gases from one vessel to another. It is simple to construct, can be adapted to meet most requirements and is readily tested. It consists of a U-tube which may be a simple line of copper tubing, or to reduce heat losses, thin walled stainless steel tube

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with wrought copper elbows. This tube is surrounded along most of its length by a metal vacuum jacket made from standard pipe or tubing. The inner tube is first made up but not formed, then cylindrical members of suitably large bore and length are slid over the inner tube which is then formed using right angle bends. Three components make up the outer jacket between two bends of the inner tube. These components consist of two equal diameter metal tubes of appropriate length which are joined by a third telescopic centre piece so that the main members can slide during assembly to give sufficient clearance to perform the bending operating on the inner tube. After bending the inner tube the jacket components are positioned and spacer discs of polythene or phenol impregnated linen are adjusted near their outer ends. The centre piece is then fixed to join them together. The corner pieces of the vacuum jacket are of brass and each is made in the form of a pill box of size suitable to the radius of the bends in the inner tube and having holes in their walls to receive the outer tubes. They are placed in position and soft soldered to the ends of the outer jacket, the box lids being added after positioning the inner tubes to avoid metal to metal contact with the jacket. A seal off valve is incorporated in each corner piece. Before adding the termination blocks an ohmmeter check should be made to ensure that there is no metallic contact between inner tube and jacket.

Note by R. B. Jacobs & R. J. Richards *Rev. Sci. Instrum.* **28**, April 1957 291-292

189/III

A Beam-Scanned Rotating Heavy-Ice Target for High Loads

Holland. From the several possible targets for the D (d, n)^{3}He reaction the heavy-ice target proves to be the most suitable for outputs of about 10^{10} neutrons per second. Beam scanning in two perpendicular directions combined with rotation of the target is found to be necessary to secure a sufficient life of the ice surface. The construction is briefly described, including the Teflon vacuum seals on the shaft driving the target.

Article by J. H. Spaa *J. Sci. Instrum.* **35**, May 1958 175-178

190/III

Light Transmissive Electrically Conducting Article

See Abstract No.: 31/III

191/III

Improvements i.a.r.t. the Production of Thin Layers

See Abstract No.: 15/III

192/III

Improvements i.a.r.t. a Process and Apparatus for the Production of Thin Deposits upon a Support by Decomposition of a Gaseous Material

United Kingdom. High stability 'cracked carbon' resistors can be produced by decomposing hydrocarbons in the gaseous state on the surface of an insulator. Difficulty is experienced in controlling the low flows of vapour required for this technique, but the patent seeks to overcome this by evaporating the hydrocarbons, preferably heptane, benzene or methane, and allowing the vapour to pass through a porous disc of a non-fibrous material, such as sintered glass, to reduce the flow. Discs in the range of 15, 30 or 40 microns are suitable. These may be mounted in tubes and, though their effect must be determined experimentally, they can be used in series—parallel arrangements, or with partial sealing of their faces to give the desired flow restriction. An oven is described for decomposing the vapour onto insulating substrates in the form of refractory rods. These rods are fed into a cylindrical refractory tube which is surrounded by an electric furnace and can be rocked about its axis to insure their even heating. The furnace tube is sealed by metal end pieces through which pipes pass. The tube is continuously evacuated through one of these and, when the temperature of the oven reaches 1,000°C, the hydrocarbon vapour is introduced through the other. To ensure an even coating, the ends from which evacuation and vapour introduction take place are interchanged several times during the deposition. Other materials than carbon may be deposited. Boron trichloride, for instance, may be decomposed to give a boron deposit. If duplicate vaporisers and flow restrictors are available, two deposits, for instance boron and carbon, may be put down at the same time.

Erie Resistors Ltd. & A. F. Drewett *Brit. Pat.* 760,328

193/III

Fabrication of Holes of about 1 μ in Diameter Used for Producing α-Particle Microbeams

United Kingdom. The problem of producing holes of about 1 μ in diameter has arisen in connection with the irradiation by α-particles of single parts of individual cells in tissue culture. Holes of this diameter are needed to collinate α-particles to beams of 1 μ–1½ μ diameter. The discs of copper normally used as collimator and objective in electron microscopes are of a diameter 3 mm., thickness 20 μ, and aperture diameter about 12 μ, which may be reduced to 6 μ by increasing the thickness of copper deposited in the electroforming process. Further reductoin of the aperture diameter by this copper-plating method is not practicable, and the holes are finally reduced to about 1 μ diameter by vacuum evaporation. The most suitable metal for evaporation onto the discs has proved to be tin. Its intermediate Atomic Number makes it neither reactive to the α-particles, nor liable to scatter them. Tin is easy to deposit by vacuum evaporation, and adheres permanently to the copper disc. The evaporating apparatus used is standard, and is designed to treat eight discs simultaneously. They are placed at least 20 cm. from the evaporating filament and are cleaned by being made one electrode in a low pressure discharge. Cleaning takes place before each evaporation, of which there may be as many as thirty in a reduction from 6 μ to 1 μ. Hole diameters are checked under a microscope after each evaporation. It appears that the holes are originally conical in shape but are filled in by the depositing beam of tin atoms, which should therefore be perpendicular to the disc surface. A properly filled-in hole will have approximately the same diameter as the emergent α-particle beam, showing that a thickness of tin sufficient to stop the α-particles (approx. 5.5 μ) has been built up.

Article by M. I. Davis C. L. Smith & P. C. Smethurst *J. Sci. Instrum.* **34**, January 1957 32

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Improvements r.t. the Treatment of Materials by Electron Bombardment — 194/III

See Abstract No.: 184/I

Ultrasonics plus Vacuum in Metal Cleaning — 195/III

United Kingdom. A brief note on an improvement in the ultrasonic technique for cleaning watch parts, etc. Working under vacuum removes dissolved air from the solvent, making cavitation more effective, and also removes air pockets from components of intricate shape allowing better contact between solvent and surface.

Note by Anon. *Electronic Engng.* **30**, April 1958 199

Testing Machine for Short-Time Creep and Stress-Rupture Testing at 2,000 to 2,500°C — 196/III

United States. Data on mechanical behaviour of materials under tensile loads is valuable to designers of high-temperature equipment, but most apparatus already available is not suitable for temperatures above 1,000°C. The equipment described in the paper enables tests to be made at temperatures between 2,000 and 2,500°C in vacuum or in a helium atmosphere. The specimen used is a bar turned to $\frac{1}{4}$ inch diam. over a length of $\frac{3}{4}$ or 1 inch in the centre. It is held vertically between loading grips made of either tungsten or graphite, inside a heater made from a seamless tube 5 inch. long, 1 inch diam. made from tantalum sheet 0.02 inch thick. This tube is mounted between tantalum electrodes carried in water-cooled copper electrode holders sealed by O-rings through the stainless steel wall of the vacuum vessel. The tube is heated by a.c. and requires 1,500 A at 8 V to reach 2,500°C. The heater is surrounded by a radiation shield made of 15 wrapped layers of 0.002 inch tantalum sheet. Vertical slots in both sides of shield and heater are aligned with a quartz window in the main vacuum vessel and enable a view to be obtained past the specimen to the opposite side of the vessel. This arrangement allows of optical pyrometer measurements or of observation or photography of the incandescent specimen, so that variations in the cross section can be followed. The load is applied by a lever to a vertical rod running through a bellows into the vacuum chamber. The chamber is evacuated by a conventional pumping system with cold traps.

Article by M. C. Smith D. M. Olson & H. L. Brown *Rev. Sci. Instrum.* **28**, July 1957 543-547

Blunting of Tungsten Needles by Surface Diffusion — 197/III

United States. Progressive blunting of tungsten needles heated in vacuum is made continuously or intermittently visible by a technique permitting needles to be heated while mounted in a conventional electron microscope. This technique permits the study of surfaces having small radii of curvature where detectable geometric changes due to diffusion occur in conveniently short periods of time. When the needle shape remains similar to its original geometry as its radius increases, a theory of Herring is applicable for distinguishing whether the transport process involved is volume or surface diffusion. Evidence is presented which indicates that surface diffusion predominates in the conditions of these experiments, *i.e.* for temperatures from 2,600°K to 2,900°K and radii from 3×10^{-6} cm. to 3×10^{-5} cm.

(*Authors*)

Article by J. L. Boling & W. W. Dolan *J. Appl. Phys.* **29**, March 1958 556-559

The Vacuum Process for Concrete on a Rockfill Dam — 198/III

United Kingdom. A vacuum process was used for the first time in the main concrete work on the Rockfill Dam which forms part of the North of Scotland Hydro-Electric Board's scheme. The dam is 1,100 feet long, has a maximum height of 110 feet and carries a 10-foot wide roadway across the top. The downstream side was battered to a slope of 1 in 1.4; the upstream face was battered to a slope of 1 in 1.3 on which a 12–15 inch. thick reinforced concrete membrane was laid. The area of the membrane is 14,800 square yards. Copper seals with bitumen filler were used as construction joints to ensure water-tightness and flexibility. The vacuum was applied to special top shutters of waterproof plywood. The edges were sealed with timber strips bedded in Bostik and the small air space required was formed by a sheet of $\frac{1}{4}$ inch mesh, a screen of fly gauze and a linen cover over the whole face. Four of these special shutters, each 20 ft. by 2 ft. were used to each 20 ft. by 20 ft. bay. By the time the fourth was in position the first was ready for stripping and was then placed above the fourth. Leap-frogging in this manner enabled a bay to be concreted in $2\frac{1}{2}$ hr. Two gangs of four men have laid five of these in 10 hr. and in the best month 99 bays were laid by the same labour force in spite of the fact that the site is exposed to prevailing west winds, driving rain and snow. Although none of the men had any previous experience of the method, they were entirely responsible for handling the shutters placing the concrete, applying and timing the vacuum, and cleaning and maintaining the vacuum shutters. A vacuum reading of 15–20 inch. Hg at the shutter was obtained by means of two rotary pumps of 348 and 275 cu. ft. per min. capacity driven by 25 and 15 h.p. electric motors and sited so as to minimise movement as the work proceeded. Tanks were fitted in the main line to collect water drawn out of the concrete. Some trouble was experienced in the winter months due to freezing of water in the pipe-lines and it was found helpful to steam-heat the aggregate and avoid opening the line to the atmosphere once the vacuum had been applied. The linen on the shutter had to be renewed after being used 20 times and the mesh and gauze after about 180 uses. The timber sealing round the edges needed frequent replacement but the plywood face of the shutter was still in good condition at the end of the job after being used about 750 times. The surface of the concrete after stripping was dry and dense and needed no special finishing. Tests from time to time during the progress of the work showed that the concrete was considerably stronger and of higher density than similar concrete laid conventionally. Millars Machinery Co. Ltd. are the exclusive licensees for the vacuum concrete plant.

Article by Anon. *Civil Engng.* Jan. 1957 55-56

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40 — GASES AND VAPOURS — 40

Ionisation Phenomena in Gases

1/IV

Italy. The third international Conference on Ionisation Phenomena in Gases, arranged at Venice during June 11–15th 1957 by the Societa Italiana di Fisica, is reviewed. The subjects covered include: Atomic and ionic collision processes, spark breakdown, high temperature plasma and pulsed discharges, arcs and glows and plasma theory.

Article by W. R. S. Garton & L. Latham *Nature* **180**, 19.10.1957 790-794

Formation of Negative Ions in Gases by Secondary Collision Processes

2/IV

United States. Experiments on the formation of negative ions by electron bombardment of hydrogen, oxygen, and water vapour at pressure of 0–4 mm. are described. The only ion observed in hydrogen is H^-, formed by dissociative attachment. Pressure dependence of the O_2 intensity shows that a secondary collision is involved in its formation. Data on the formation of O^- and O_2^- at low electron energies indicate that the secondary collision leading to the formation of O_2^- does not involve an O^- ion. The formation of O_2^- probably involves a stabilising collision with a molecule of gas after the initial formation of the ion by electron attachment. From the behaviour of H^-, O^-, and OH^- intensities with H_2O pressure, it is shown that the secondary process for the formation of OH^- in water vapour is probably

$$H^- + H_2O \longrightarrow OH^- + H_2$$

The cross section for this reaction is more than 10^{-17} cm.^{2}.

(*Author*)

Article by E. E. Muschlitz Jr. *J. Appl. Phys.* **28**, Dec. 1957 1414-1419

Measurement of Low Vapour Concentrations by Collision with Excited Rare Gas Atoms

3/IV

United Kingdom. High, steady concentrations of excited atoms can be established in a rare gas by the simultaneous application of a high-intensity electrical field and a source of free electrons. The acceleration of the electrons in the field raises their temperature to excite the rare gas by collision. The introduction of vapour molecules into a volume of rare gas containing excited atoms leads to an increase of the ion concentration. If the gas is contained in an ionisation chamber the concentration of vapour molecules can be measured either in terms of the increase of ionisation current or the decrease of potential across the chamber.

Letter by J. E. Lovelock *Nature* **181**, 24.5.1958 1461-1462

'Clean-Up' of Radioactive Gases Used for Surface Studies

4/IV

See Abstract No.: 24/I

Permeation of Gases through Solids

5/IV

United States. The paper reports investigations carried out on the permeation of gases through solids with particular reference to the limitations which this process imposes on the degree of vacuum obtainable in a system. In the experimental work great emphasis has been given to the necessity for identifying the gases appearing on the low pressure side in order to avoid misleading results. A mass spectrometer has been used in the experiments. Data is given for the permeation of gases, particularly helium, through glasses of different compositions. Appreciable permeation rates have been observed for helium, hydrogen, deuterium and neon but the rates for gases of greater molecular diameter (> 3 Å) is exceedingly small. With change of glass composition, the rates of helium permeation vary over a million fold. Measurements at different temperatures show that the permeation constant K is related to temperature by the expression $K = A\, e^{-\frac{Q}{RT}}$ where Q is the activation energy per gram atom and involves not only the diffusion process but also the adsorption and solution processes. In the discussion of gas permeation through metals it is pointed out that no rare gas permeates through any metal. Other gases show great specificity; their rates vary as the square root of the pressure, implying that the gas dissociates at the surface. Gases may enter the metal not only from the gas phase, but also through other external processes such as corrosion, acid attack and electrolysis. Thus, water-cooled steel vacuum devices may have the vacuum spoiled by hydrogen permeation from corrosion at the water-steel outer face. Gas permeation through organic polymers is reviewed. It is stated that all gases go through all polymers and that the permeation rate is proportional to pressure; hence the molecules enter the solid undissociated.

Article by F. J. Norton *J. Appl. Phys.* **28**, Jan. 1957 34-39

Helium II Film Filter

6/IV

Canada. The construction and properties of a wire filled tube filter for liquid helium II film is described. It consists of a copper-nickel tube of 0.272 cm. i.d. containing 1,200 Eureka wires each of length 36 cm. and 0.002 inch diam. The assembly is then drawn down through steel dies of progressively decreasing diameter until the diameter of the filter has been reduced to about two-thirds of its original value. It is found that a logarithmic plot of the outer diameter of the filter tube *versus* the channel width is a straight line after close packing of the wires starts, the channel width being determined by measurement of the rate of flow of helium gas through the tube, or directly from microphotographs of a cross-section of the tube. Reference is made to the type of flow—viscous or superfluid—which is obtained with different channel widths for liquid helium II.

Article by K. L. Chopra *Rev. Sci. Instrum.* **28**, February 1957 146-147

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Vacuum Flow of Gases Through Channels with Circular, Annular and Rectangular Cross Sections

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8/IV

High Sensitivity Mass Spectrometer for Noble Gas Analysis

See Abstract No.: 82/II

9/IV

Thermal Transpiration in the Mass Spectrometer Inlet Systems

See Abstract No.: 81/II

10/IV

The Mechanism of Water Vapour Movement in Porous Materials

Germany. The work reported here forms an extension of the work carried on by Volmer on the permeability of paper and foils to water vapour. The present study is concerned with plastic foil, parchment paper, etc. In addition, the temperature dependence of surface diffusion has been measured and aspects of this are discussed.

Article by H. Schaschek *Chem. Ing. Tech.* **28**, Nov. 1956 698-702

11/IV

Equilibrium Between Glass and Water Vapour at Bake-Out Temperatures

See Abstract No.: 27/I

12/IV

Reaction Between Oxygen and Hot Gold

See Abstract No.: 21/I

13/IV

Thermal Effects in the Chemisorption of Oxygen on Nickel: A Comparative Investigation Using Powders and Evaporated Films

See Abstract No.: 57/III

14/IV

Hydrogen Sorption and the Parahydrogen Conversion on Evaporated Nickel Films

See Abstract No.: 172/I

15/IV

The Hydrogen Content of Atmospheric Air at Ground Level

United Kingdom. There are wide divergencies in the published figures of the hydrogen content of the atmosphere ranging from the 0.5 parts per million given in the Smithsonian Physical Tables 1954, to the 100 parts per million in the Handbook of Physics and Chemistry 1956–7. The method described for separation and determination of hydrogen was originally used for the determination of krypton and xenon content of the air. A demountable metal still consists of a boiler connected by vacuum-tight joint to a fractionating column and reflux section. The boiler is surrounded by a Dewar vessel and the reflux section is mounted in the centre of a vacuum chamber. The still is evacuated and taken to a suitable location for collection of the air sample; the Dewar and the vacuum chamber are filled with liquid nitrogen, and air is allowed to flow in for about ½ hour. Precautions are taken against contamination of the air and it enters over a suitable drying agent. The still is connected to the rest of the apparatus in the laboratory and the gas sample allowed to fractionate to separate the higher boiling point gases, reflux being accomplished by the drop in temperature resulting from the pumping of liquid nitrogen from the vacuum chamber. The hydrogen-nitrogen mixture is passed over heated copper oxide, the water vapour condensed in a liquid-oxygen trap in a modified and calibrated McLeod gauge and the remaining gas pumped to the meter. The water is free from contaminating gases by pumping the gauge down to 10^{-4} mm. Hg. Measurement of the vapour pressure over ice checks that only water vapour is present. Since the accuracy of the method in its present form is not better than $\pm$ 10% as shown by checks on prepared samples and further work needs to be done, the results cannot be taken as conclusive. It would seem however that the hydrogen content of the atmosphere at ground level varies with prevailing weather conditions as 26 analyses gave values from 0.13 to 1.99 parts per million with a mean value of 0.6 parts per million.

Article by E. Glueckauf & G. P. Kitt *A.E.R.E. Quarterly J. of The Roy. Metereol. Soc.* October 1957 522-528

16/IV

A Controllable Source of Low-Pressure Hydrogen

United Kingdom. The device consists of a nickel-gauge cylinder (100 mesh) mounted on an alumina-coated heater. The active material is zirconium powder (300 mesh) suspended in acetone and mixed with a nitrocellulose binder, this is brushed on to the gauge. The assembly is mounted on a two-wire glass pinch. The nominally pure zirconium contains sufficient quantities of hydride but additional activation can be carried

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out by filling the system with hydrogen and raising the temperature gradually to 400°C. The system is baked for one hour at 450°C and the heater is flashed at 1,000°C for a few seconds. Data are given showing the hydrogen pressure produced in a sealed-off system at various heater voltages; they range from below 0.001 to 0.150 mm. Hg. It is claimed that the device provides a more accurate and reliable means of controlling the hydrogen pressure than a needle valve and hydrogen reservoir. Moreover, the zirconium acts as a getter for oxygen, nitrogen and carbon dioxide.

Note by A. E. Barrington & F. T. Turner *J. Sci. Instrum.* **34**, July 1957 286-287

Handling Compressible Fluids in Chemical Processing
See Abstract No.: 165/II

17/IV

The Shock Tube—Just a Piece of Pipe
See Abstract No.: 180/I

18/IV

41 — FREEZE DRYING — 41

Mass Transfer at Low Pressures
See Abstract No.: 141/I

19/IV

Heat and Mass Transfer in Freeze Drying

20/IV

United States. An investigation was made into the effect of operating conditions, *e.g.* rate of energy input, vacuum, and condenser temperature on the drying rate and temperature level of various substances in a simple radiant heated tray freeze drier. Theoretically, the rate of drying should be dependent on both heat and mass transfer. For water ice sublimation, the sublimation rates varied from 0.5–1.5 lb/sq. ft. hroat pressures of from 185–2,150 μ. The water vapour pressure at the interface was approximately equal to the total pressure which confirmed the theory that interfacial mass transfer is not an important effect at pressures over 150 μ. It was shown that water vapour diffusion is the most important means of mass transfer to the condenser. In the drying of materials which contain solids, the mass transfer resistance of the cake layer becomes of considerable importance. Slab drying rates averaged 0.01–0.30 lb/sq. ft. hr., but the rate was less for material in the form of pellets. Substances investigated for product quality were: (*a*) whole milk, (*b*) baker's yeast, (*c*) a bacterial suspension. Slow drying was found to be preferable in the first two cases, but freezing and drying rates were not important with bacterial suspensions.

Dissertation by J. B. Lambert *Dissertation Abstracts* **16**, 1956 2116

Improved Heat Transfer System for Freeze Drying

21/IV

United States. As compared with flat-tray design, ribbed-tray design with a given load yields greater output at any usable temperature condition tested. In addition, ribbed design shows more rapid increase in output as heating intensity is increased. Actual metallic tray heating surface to which material was exposed in ribbed trays was approximately 215% greater than in flat trays.

Article by I. M. Abelow & E. W. Flosdorf *Chem. Engng. Prog.* **53**, 1957 597-600

The Freezing of Water Droplets
See Abstract No.: 137/I

22/IV

The Loss of Inorganic Non-Volatile Substances During Freeze Drying
(Reference only)

23/IV

Article by K. Neumann *Forschungsber Wirtschafts-u. Verkehrsmin. Nordrhein-Westfalen* **189**, 1955 35-39

Automatic Recording of Weight and Temperature for Vacuum Sublimation Studies

24/IV

United States. In work concerned with the vacuum drying rates of frozen biological specimens the authors have designed a drying apparatus which enabled continuous records to be made of both weight and temperature. The drying chamber is a glass tube 130 cm. long and 5.5 cm. diam. which can be opened at a joint 50 cm.

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from the bottom to give access to the specimen and is pumped *via* a side arm near the top of the tube. The system is mounted on a drill press column using two supports so as to give rotational adjustment in any direction. These supports can then be raised or lowered without disturbing the orientation of the system. The optical system is used to give continuous weight records. The glass tube is vertical and the sample is placed on a scale pan hanging from a special alloy spring (Cenco N.7508) carrying a cross hair at its lower end. The spring extension is 5 cm./gm. and copper wires are used to suspend the spring and to suspend the copper scale pan below it. A fine coil of 0.001 mm. constantan wire is connected to the base of the scale pan. The junction between this wire and the scale pan serves as the variable temperature junction of the copper constantan thermocouple. The cold junction is made outside the tube and the copper and constantan leads are taken out of the vacuum system *via* a fine capillary tube sealed with vacuum wax and situated at the top of the glass tube. The emf's introduced by the two copper alloy junctions (1.9 $\mu\mu$/°C) are only significant in the most precise work. Continuous temperature records are made using an amplifier and recorder. Light from a tungsten strip filament is condensed on the horizontal cross hair below the spring, and a telemicroscope fitted with a Dove prism serves to project a vertical image of the cross hair *via* a cylindrical lens onto a horizontal scale on a fine grain positive film 12 cm. wide which is moved vertically at constant speed. Time lines are made on this film at one minute intervals by using a rotating shutter driven by a synchronous motor. Light intensity is controlled by supplying the source lamp from a variable transformer. It is possible to estimate 0.1 mm. on the film; which corresponds to 0.002 mm. extension of the spring, *i.e.* to a specimen weight change of 40 mg. The system is suitable for samples between 20 and 500 mg. in weight.

Note by J. L. Stephenson G. W. Smith & H. V. Trantham *Rec. Sci. Instrum.* **28**, May 1957 381-382

25/IV **Advances in Methods and Applications of Freeze Drying**

See Abstract No.: 60/I

26/IV **The Separation of Human and Bovine Plasma Thromboplastin with Ether and a Study of Its Properties**

United Kingdom. Plasma defibrinated under special conditions was treated in the cold with ether to precipitate crude thromboplastin. After dialysis against cold water and freeze-drying, the nitrogen content was 16.1% for human material and 13.7% for bovine. Acid hydrolysis revealed the same amino acids in both, namely, aspartic acid, glycine, serine, glutamic acid, alanine, threonine, leucine(s), valine tyrosine, lysine, and cysteine and its decomposition products. The product rapidly lost activity when dissolved in saline and it was very sensitive to changes in pH. Concentrated solutions of bovine and human thromboplastin clotted citrated plasma under specified conditions, in 5 and 7 sec. respectively.

(*Chemical Abstracts*)

Article by F. Nour-Eldin & J. F. Wilkinson *J. Physiol.* **132**, 1956 164-172

27/IV **Lyophilised Plasma**

France. The effect of freeze-drying on the lipoproteine of normal and lipomic human plasma has been studied

Article by M. Burstein & J. Samaille *Rev. Hemat.* **11**, 1956 96-100

28/IV **Preservation of Leptospirae by Drying**

United Kingdom. Preservation of three strains of leptospirae by drying, a method hitherto unsuccessful, has now been achieved using a vacuum evaporation technique. The organisms were prepared in the form of concentrated suspensions, one drop volumes of which were used to inoculate sterile frozen-dried plugs (0.25 ml.) of starch-peptone. The tubes containing the plugs were exposed immediately to high vacuum and P_2O_5, when the viscous solution which formed evaporated rapidly to give a dried foam. After about 5 hours evacuation the tubes were constricted and sealed. Recovery of the organisms is possible after 24 months storage at room temperature. Details of the method of recovery are given.

Paper by D. I. Annear *J. Path. Bact.* **72**, 1956 322-323

29/IV **Near-Eastern Equine Encephalomyelitis**

Syria. A preliminary report is given of an investigation made by the Food and Agriculture Organisation Technical Mission to Syria, into encephalitis of horses and donkeys in Egypt and Syria. The virus isolated from outbreaks in sheep and cattle seems to be indistinguishable from that found in the equine disease. The symptoms are briefly described, as are experiments in which chicken, kitten, rabbits and sheep were inoculated with the virus. The virus is reported to pass readily through various filters and membranes. It can be preserved for at least a year at −30°C as frozen tissue and freeze-dried specimens were still viable after 3½ years at −30°C. There is a brief discussion of the possible part played by ticks in the transmission of the disease.

Report by R. Daubney & E. A. Mahlau *Nature* **179**, 16.3.1957 584-585

30/IV **This Unit Puts Freeze-Drying Right on the Processing Line**

See Abstract No.: 186/III

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Freeze-Dried Food

United States. This article refers to experiments on the freeze-drying of food, carried out by Dr. A. Copson of Raytheon Manufacturing Co. Conventional techniques are used except that the heat of sublimation is supplied by eddy-current heating in order to obtain uniform heating throughout the material. Foods so treated lose up to four-fifths of their weight and can be stored in plastic envelopes filled with nitrogen.

31/IV

Article by Anon. *Time* 20.5.1957 55

Storing Freeze-Dried Beef

United States. When beef is freeze-dried, removal of oxygen converts the oxymyoglobin to myoglobin which subsequently oxidises to unwanted metmyoglobin during storage at 100°F. This, and other undesirable reactions, can be prevented by storing in a vacuum or an inert atmosphere.

32/IV

Abstract by Anon. *Food Engng.* **28**, July 1956 153 Orginal Article in *Food Research* March-April 1956 195-205

Management of an Artery-Graft Bank

See Abstract No.: 186/I

33/IV

45 — ELECTRONICS — 45

Production of High Temperatures and Nuclear Reactions in a Gas Discharge

United Kingdom. A report from A.E.R.E., Harwell, on the apparatus known as 'Zeta'. There is a brief description of the apparatus, with its ring-shaped discharge tube, of 1-m bore and 3-m mean diameter, formed of 1-inch thick Al, which links the iron core of a pulse transformer, through the primary of which a bank of capacitors, storing up to half a million joules, can be discharged. The tube contains gas, usually at a pressure of about 10^{-4} mm. Hg, made weakly conducting by an r.f. discharge, the ionised gas then forming a single-turn secondary to the pulse transformer. A unidirectional pulse of up to 200kA, lasting several m.sec, is produced by the discharge, and completely ionises the gas. The discharge has a natural tendency to constrict itself away from the containing walls (the 'pinch effect'), but is only made reasonably stable by the use of an axial magnetic field, which is provided (up to 400 gauss) by coils wound over the toroidal tube. The discharge can be repeated every 10 sec, and studies made of the spectra of the gases used and of neutron yield from nuclear reactions. Temperatures of the order 1–5 × 10^6 deg K were estimated from the Doppler broadening of spark lines and from neutron emission and from the D–D reaction when deuterium (plus some O_2 and N_2) was used. Streak photographs of a discharge using He confirmed the constriction of the discharge and led to the estimate that the diameter of the current channel was between 20 and 40 cm, in the 100 cm. diameter tube.

34/IV

Article by P. C. Thonemann E. P. Butt R. Carruthers A. N. Dellis D. W. Fry A. Gibson G. N. Harding D. J. Lees R. W. P. McWhirter R. S. Pease S. A. Ramsden & S. Ward *Nature* **181**, 25.1.1958 217-220

Electrostatic Charge Separation at Metal-Insulator Contacts

United States. Studies have been made of the electrostatic charge produced in vacuum on inorganic insulators put in rolling contact with metal surfaces. Investigations aimed at (*a*) extending earlier work to include a variety of insulators and metals and (*b*) clarifying the mechanism of the charging process when carried out in a vacuum. The insulators were 6 mm. diameter optically polished spheres. These were placed inside large magnetically-driven rotating barrels of Ni, Cu, or Pt. The charge developed on the sphere was sensed by an electrostatic probe. The entire system was enclosed in a Pyrex envelope which could be evacuated by a liquid N_2–trapped mercury diffusion pump. After overnight bake-out at 350°C pressures of 2 × 10^{-9} mm. Hg could be attained by combined ion and diffusion pumping. Of the insulators used, quartz gave the most reproducible results. Spheres of both fused and mono-crystalline quartz were used. Both charged negatively in an almost identical manner. Little change in charging characteristics was noted if chemically cleaned air-dried quartz was outgassed at ~700°C and 3 × 10^{-9} mm. Hg. Saturation of charge was greater for spheres with a smoother finish. The charging mechanism for quartz is thought to be transfer of electrons. Thus quartz, having an electron affinity or 'effective work function' larger than the metal accepts electrons at the point of contact. The charge measured is considered to be less than that originally produced due to field emission-induced back leakage of electrons during separation of surfaces. The electric field at the charged quartz surface is estimated to be of the order of 5 × 10^5 V/cm. The other insulators tested were single crystals of Al_2O_3, MgO, NaCl, KCl, KBr and KI. These charged less vigorously and less reproducibly than quartz. No evidence of electron transfer was found and ion transfer mechanisms are suggested. The charge distribution on Al_2O_3 spheres was markedly dependent on crystallographic orientation. MgO containing either excess Mg or O as bulk impurity charged positively against nickel, in a uniform manner. Stoichiometric MgO charged negatively. Baking of MgO containing O in vacuum at 1,200°C removed O and reversed the sign on charging. Polishing for one hour of the baked sphere caused it to charge positively again. Alkali halides always charged positively, but reproducibility was poor.

35/IV

Article by P. E. Wagner *J. Appl. Phys.* **27**, Nov. 1956 1300-1310

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36/IV

Letter by W. P. Baker *Nature* 181, 21.6.1958 1726

Influence of the Ambient Medium on the Breakdown Voltage of Sheet Insulating Materials

United Kingdom. The breakdown voltage of films of cellulose triacetate and polyethylene terephthalate was measured at 50 c/s in air and inert gases at pressures ranging from rough vacuum up to 8 atmospheres. The tests were made at room temperature using a sphere/plane electrode system and a step-by-step voltage application. It was found that the breakdown voltage of the film specimen increased with gas pressure, and curves show that the electric strength was also influenced by the nature of the gas. The highest electric strength was obtained with helium, and the lowest with argon.

37/IV

Article by N. B. Rosanova & V. L. Granovskii *Soviet Phys. Techn. Phys.* 1, 1957 471-478

On the Initiation of Electrical Breakdown of a High Vacuum Gap

Russia. Investigations over many years into the mechanism of breakdown of a high vacuum has established that the final stage of the process consists of an arc discharge in the vapour of the material of the electrodes. This investigation is concerned with the initial stages of the breakdown process where there is still no unanimity of opinion as to the precise mechanism. Some workers in the field have suggested that evaporation of the material of the anode, heated by electron bombardment, is the important process while others have assumed that ions are ejected from the anode by electron bombardment. Attention has also been drawn to the possible role played by x-radiation from the anode in producing additional electron emission from the cathode. In this investigation the discharge was struck between a hemispherical cathode and a plane anode, each about 1 cm. in diameter, placed at various distances up to 3 mm. apart in a glass envelope evacuated to a pressure less than 10^{-5} mm. Hg. Measurements of both static and pulsed breakdown voltages were observed for Al, Cu, Fe, Ni, Mo, W and graphite electrodes. It was found that the breakdown voltage for gaps which differ only in the material of the anode increases in the following order: graphite, Al, Cu, Fe, Ni, Mo, W. This sequence is not altered if the length of the gap or the cathode material is changed. Moreover, this increase in the electrical breakdown strength proceeds parallel to the increase in the mechanical strength of the anode material if the latter is characterised by Young's modulus. Measurements of the x-ray intensity by an ionisation chamber showed that the electrical breakdown strength of the gap does not decrease (as had previously been suggested) but increases with an increase in yield of x-radiation in the pre-breakdown stage. It is therefore suggested that the x-radiation is a factor which accompanies the occurrence of breakdown, but which is not its cause.

38/IV

Article by W. K. Mansfield & R. L. Fortescue *Brit. J. Appl. Phys.* 8, February 1957 73-78

Pre-Breakdown Conduction between Electrodes in Continuously-Pumped Vacuum Systems

(Reference only)

39/IV

Letter by G. B. Walker & E. L. Lewis *Nature* 181, 4.1.1958 38-39

Vacuum Breakdown in Dielectric-Loaded Wave-Guides

United Kingdom. An investigation of the breakdown properties of dielectric discs used for loading wave-guides to obtain a slow wave structure is reported. Titanium dioxide ceramic is a promising material for this purpose since it has a loss tangent of only 0.0003 and a dielectric constant of 90 at 3,000 Mc/s. In these experiments a resonant cavity 20 cm. long was loaded with four such discs arranged at half-wave intervals. The cavity was excited in an E-mode by a 2 MW. magnetron operating at 3,000 Mc/s, the electric field being most intense along the axis. The field was normal to the discs, the maximum value of its radial component occurring inside each disc at its mid-plane. It was found that breakdown was liable to occur at an axial field strength of less than 10^5 V/cm. and a pressure of 10^{-5} mm. Hg.; breakdown was accompanied by a sudden increase in x-ray output from the cavity. The disc sustained superficial damage, the titanium dioxide being reduced to a lower semi-conducting oxide. A photograph shows the tracks produced on the discs after breakdown. A much higher breakdown strength was observed with discs coated with a layer of glaze (consisting of green resistor enamel which is mainly lead borate) of a thickness of a few thousandths of an inch. No breakdown occurred at 3×10^5 V/cm. which was the highest field strength attainable.

40/IV

Article by D. T. Rose & S. C. Brown *J. Appl. Phys.* 28, May 1957 561-564

Microwave Gas Discharge Breakdown in Air, Nitrogen and Oxygen

United States. The breakdown field for pure air lies between those for nitrogen and oxygen. A calculation of the high-frequency breakdown field in air is made from d.c. data, using the measured Townsend ionisation coefficient, electron attachment coefficient, and average electron energy. The present data agree well with this calculation.

41/IV

Improvements r.t. the Treatment of Materials by Electron Bombardment

See Abstract No.: 184/I

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An Electron Gun Alignment and Mounting Technique
(Reference only)

42/IV
Note by
L. C. Robinson
J. Sci. Instrum.
35, March 1958
112

Slalom Focusing

43/IV

United States. In the search for a scheme of electron focusing which would permit a beam to weave in and out through an r.f. structure, it was discovered that a linear array of line charges in free space produces two equipotential surfaces which contain exact electron trajectories. The field about such an array may be nearly duplicated by an array of positive wires sandwiched between two negative plates. It has been found that such a structure will effectively focus a ribbon-type electron beam of surprisingly high perveance. A backward-wave oscillator was built which utilised this 'slalom' type focusing. A beam tester was also constructed which has a beam-focusing structure just 10 times the size of that of the oscillator: 97% transmission through 27 wires of a beam of perveance (2×10^{-6}) has been achieved with this tester. Four different types of electron gun used in the experiments are illustrated.

Article by
J. S. Cook
R. Kompfner &
W. H. Yocom
Proc. I.R.E.
45, Nov. 1957
1517-1522

On Internally Metallising a Betatron Toroid by Vacuum Deposition

44/IV

United Kingdom. The general method of producing a conducting metallic film on the internal surface of apparatus is illustrated by a description of the internal coating of a betatron toroid. Two parallel wires plated with chromium and concentric within the toroid were used for the main section, which was coated in two halves. Ports were coated using a conical wire basket containing chromium, the wire acting as the filament. The toroid was mounted in an oven, and was connected to the vacuum system through the oven floor. Temperature distribution was checked using thermocouples. Sealing was achieved using a silicone gasket between flanges clamped with split rings. The toroid was cleaned thoroughly, the filmaents inserted, and the whole pumped to 10^{-4} mm. Hg and heated to 350°C. The filaments were then fired, and the apparatus cooled slowly. The process was repeated for the other half of the toroid. A hard uniform coating of a resistance of 56 ohms per square was obtained.

Article by
K. R. Allen
F. Ashworth &
G. Siddall
J. Sci. Instrum.
33, Nov. 1956
445-446

The Cern 600 MeV Synchro-Cyclotron

45/IV

Europe. Describes the new machine built by the European Organisation for Nuclear Research, CERN, at Geneva. This delivers 600 MeV protons and is the third largest in the world. It is hoped to obtain a proton current of 1 or 2 mA, in pulses of a few hundred microsec duration, repeated at 55 p.p.s. The vacuum chamber is made of stainless steel, and is pumped by two diffusion pumps each rated at 12 m.³/s. at 10^{-4} mm. Hg backed by three mechanical pumps handling 170 m.³/hr., each. The pressure obtained in the tank is said to be 3×10^{-6} mm. Hg, increasing to 6×10^{-6} mm. Hg with the ion source operating. Several photographs show the building, the magnet, the pumps and the control room, while a diagram shows the layout of the machine, and the possible emergent beams which can be used. Removable sections in the concrete shielding walls, allow the direct proton beam to enter an adjacent experimental room, or it may be deflected by a magnet into a second room, or it may be allowed to strike one or more targets and produce π mesons for experimental work. A neutron beam is also available. The installation has cost about £2,000,000 and has a staff of 30–40 physicists. Teams from member countries of the organisation will also work there

Article by
A. Lundby
Discovery
19, February 1958
56-59

Tandem Particle Accelerator

46/IV

United States. Description of an accelerator to be constructed by the United Kingdom Atomic Energy Authority.

Note by
Anon.
Engineer
204, 12.7.1957
59

25,000 MeV Particle Accelerator at Brookhaven National Laboratory

47/IV

United States. A 50 MeV linear accelerator is to be used to inject protons into the 25,000 MeV 'Alternating Gradient Synchrotron' being built at the Brookhaven National Laboratory. This synchrotron consists of an 842 ft. diameter annular steel and copper vacuum chamber, and employs 240 focusing magnets and 12 r.f. accelerating stations. It provides a pulse of 1,000 million particles every three seconds, and should be ready in 1960.

Note by
Anon.
Engineer
203, 29.3.1957
507

Description of a Simple One Million Volt Electrostatic Generator

48/IV

United Kingdom. The desire to extend certain electron scattering experiments in the energy range up to one million electron-volt prompted the construction of an electrostatic generator in the Physics Department of the University of Leicester. This machine has proved so useful and reliable that a brief description of its construction, operation and applications may be of interest to other laboratories with small financial and workshop resources.

(*Authors*)

Article by
E. Matsukawa
P. K. Rose &
J. E. Wilson
Research
10, 1957
280-282

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49/IV

Letter by D. Aitken & R. E. Jennings *Nature* 181, 21.6.1958 1726

The Microtron

United Kingdom. The microtron under construction at University College, London, W.C.1 is now operating and can accelerate electrons to 29 MeV. The poles of the electromagnet are 80 inch. dia, and they form the top and bottom of the vacuum chamber. The proposed experimental programme includes the study of bremsstrahlung spectra and the determination of the rate of energy loss of electrons in various materials.

50/IV

The Use of Getters in Magnetrons

See Abstract No.: 22/I

51/IV

Letter by M. A. Cayless & A. D. Forster-Brown *Brit. J. Appl. Phys.* 8, September 1957 380

The Diffusion of Barium in Discharge Tubes

(Reference only)

52/IV

Barium Getters and Oxygen

See Abstract No.: 20/I

53/IV

Letter by R. N. Bloomer *Nature* 179, 2.3.1957 493

Oxidation of Barium: A Confirmation of Mott's Theory of Oxidation

United Kingdom. N. F. Mott's ionic theory of the oxidation of metals has been confirmed by measurements of the rate of taking up of oxygen by barium films. The theory supposes that positive metal ions and electrons diffuse from the metal through the oxide, the metal ions moving *via* interstitial positions in the oxide lattice. At the free surface of the oxide, these ions combine with adsorbed oxygen. In this way the thickness of the oxide layer is increased. In oxide films less than about 50 Å thick the movement of ions is influenced principally by the strong electric field occurring across the oxide due to the contact potential difference between the metal and the adsorbed oxygen. In thicker films ion movement and hence oxide growth is influenced chiefly by temperature. The theory predicts that, whatever the thickness of oxide, the movement of ions becomes controlled by temperature when this is raised above a critical value, so that above this temperature oxidation should continue until all the metal has been oxidised. Below the critical temperature oxide growth stops at a limiting thickness, which should vary with temperature when this is less than the critical value. The critical temperature of barium was found to be 35°C and the results of measurements on growth rate and limiting film thickness were in agreement with Mott's theory. At 20°C a film 25 monolayers thick (equal to about 50 Å)was formed. The speed of pumping of oxygen per unit area of barium film increased with temperature only above 35°C, but the actual speeds were merely 1/25 of what they would have been if every oxygen molecule striking the surface had been compounded. From this it is concluded that the speed of pumping of oxygen by barium is limited by the superficial density of interstitial barium in the oxide. Mott's theory does not cover the form of oxidation but only further thickening of the layer. During the formation of the first monolayer the speed of pumping increased in proportion to the square root of the exposure time of the barium to oxygen, and was independent of temperature. These results are explained by the idea that oxidation starts at nucleation centres in the barium surface to form a complete monolayer. Thereafter the speed remains constant, and the oxidation proceeds in the manner described by Mott's theory.

54/IV

Paper by L. Jacob *Proc. Phys. Soc.* 70B, 1957 235-239

The Electron Bombardment of Thin Barium Films

United Kingdom. Experiments are described which show that it is possible to transfer barium from an anode as a thin film to a cathode by simple electron bombardment. This effect appears to occur with the anode cold. The 'threshold' bombarding energy is about 300 V for barium; the corresponding current density at the target is of the order of 1 mA cm.$^{-2}$. The gradual build-up of thin film activity of a pure strontium oxide cathode was followed with time. The final result was an emitter whose performance matched that of the standard BaO/SrO cathode in every way; the thin film emitter, however, exhibits a large temperature dependence but has high tenacity.

(*Author*)

55/IV

On the Mechanism of Operation of the Barium Aluminate Impregnated Cathode

United States. Emission and evaporation characteristics of a porous tungsten cathode impregnated with the composition $5BaO.2Al_2O_3$ are presented and are interpreted in terms of the cathode mechanism. Barium

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necessary for activation is generated by the reaction:

$$\tfrac{2}{3}\ BA_3Al_2O_6 + \tfrac{1}{3}\ W = \tfrac{1}{3}\ BaWO_4 + \tfrac{2}{3}\ BaAl_2O_4 + Ba,$$

and is transported through partially clogged pores, the length of which increases with the time, predominantly *via* Knudsen flow. During transport, oxygen is acquired from the tungsten, leading to a substantial content of BaO in the evaporant. The $BaAl_2O_4$ component of the impregnant is inert. Emission is substantially lower than that of an L-cathode, presumably because of release of poisoning agent accompanying the activator.

(*Authors*)

Article by E. S. Rittner, W. C. Rutledge & R. H. Ahlert *J. Appl. Phys.* **28**, Dec. 1957 1468-1473

Dispenser Cathodes

56/IV

Holland. The L-cathode developed in the Philips laboratory at Eindhoven several years ago consists of a porous tungsten body, the emitting surface of which is covered by a layer of barium and oxygen atoms. The continuous evaporation of barium during operation of the cathode is compensated by the dispensation of barium from a compound located in a chamber behind the porous tungsten body. The L-cathode can be operated continuously at very high current densities, its metallic emitting surface is robust and can be made to narrow dimensional tolerances and with good finish; it has a very long life (several thousands of hours), it recovers easily from poisoning and it is highly resistant to bombardment by ions. The application of the L-cathode in tubes, however, is limited by the relatively long time required for degassing and activation, by the difficulties in hermetically sealing the chamber containing the barium dispensing compound and by its limitations in size. Investigations in the Philips Laboratories at Irvington have led to the development of two new types of dispenser cathodes, working on the same principle as the L-cathode (and therefore having the same general electron emission characteristics) but avoiding its drawbacks. The first type is a pressed cathode, made by compacting a mixture (ratio 9 : 1 by weight) of powdered tungsten-molybdenum alloy and barium-calcium aluminate, and heating the pressed mass. The molybdenum content of the alloy (75% by weight) is essential to lower the evaporation rate of barium and barium oxide to the desired level. The second type is an impregnated cathode, made by impregnating a porous tungsten body with molten barium-calcium aluminate. Suitable dispensation of barium is obtained by using tungsten with a porosity of 17% (the porosity is 27% for the tungsten body of the L-cathode and 40% for the alloy in the pressed cathode). This porosity value also permits the application of a special technique for machining the tungsten, so that the impregnated cathode can easily be made in a large variety of shapes and sizes, including very small ones. Both the pressed and the impregnated cathode have much shorter degassing and activation times than the L-cathode and can be manufactured more readily. A pressed cathode of flat shape especially lends itself to mass production, but mass manufacture of the impregnated cathode likewise does not involve any great difficulties, while the latter type offers by far the greatest latitude in design. The calcium content of the aluminate in both types of cathodes enhances the electron emission considerably, by a mechanism which is not yet fully understood. These cathodes can also be exposed to the air and reactivated after restoring the vacuum several times during their life. The electron emission of all three types of dispenser cathodes (L-cathode, pressed and impregnated cathodes) makes them suitable for use in tubes which call for high current densities, either continuously or in pulses, for several thousand hours.

(*Authors*)

Article by A. Venema R. C. Hughes P. P. Coppola & R. Levi *Philips. Tech. Rev.* **19**, No. 6 1957/58 177-190

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Measurement of Instantaneous Absolute Barium Evaporation Rates from Dispenser Cathodes

57/IV

United States. A simple procedure for determining the 'instantaneous' absolute evaporation rate of barium from dispenser cathodes is presented. The method entails exposing a clean tungsten wire to a stream of barium and noting the time required to reach maximum emission. The sensitivity of the wire as a detector of total barium is established on an absolute basis by chemical calibration. It has been found that the wire sensitivity is a function of the barium oxide content of the evaporant stream. This fact may be employed to determine the oxide composition of the evaporant from wire emission-time measurements and a single chemical analysis. It is also possible to obtain approximate, instantaneous values of total barium and the fraction BaO at any time during tube life from wire emission-time measurements only. Calibration data are presented for almost the full range of Ba–BaO compositions.

(*Authors*)

Article by W. C. Rutledge A. Milch & E. S. Rittner *J. Appl. Phys.* **29**, May, 1958 834-839

Studies on the Mechanism of Operation of the L-Cathode I

58/IV

United States. The performance of the L-cathode has been investigated. Measurements are presented of emission, evaporation rate and nature of evaporated material, and observations on the nature of the emitting surface are reported. Emission measurements were made with a planar diode, using a guard ring round the cathode. In the space-charge region, data agreed well with the modified form of Childs' law, and discrepancies in the Schottky region were traced to the effect of surface roughness. The work function was found to be 1.67eV (extrapolated to O°K). Accelerated life tests at 1,250°C, during which emission measurements were made at 1,190°C, showed that life is enhanced by reducing the porosity of the tungsten plug and increasing the charge of oxides. It was also found that $SrCO_3$ prolongs the life over that obtained with $BaCO_3$ alone. The evaporation of Ba and BaO was followed by measuring the emission from a tungsten wire mounted near the cathode and protected when required by a shutter. The wire was operated at only 675°C to reduce the evaporation from it, and was chemically calibrated as an absolute detector of Ba. Polarographic analysis of the material evaporated from a cathode containing $BaCO_3$ only, showed Ba to be the only metal evaporated, but from chemical and adsorption analyses it is concluded that the evaporant contains a large proportion

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of BaO. The distance which adsorbed Ba atoms (or BaO) might be expected to migrate on a W surface was estimated by allowing Ba to deposit on a tungsten ribbon pierced with a narrow slot. The atoms migrated round the edges of the slot to produce a small emitting area on the other side, the emission from which gave a measure of the distance travelled by migrating atoms. The lifetime of the adsorbed atoms was obtained from the same experiment by observing the time taken for the emission to build up. The results show migrations over a few tenths of a millimetre, very much greater than the average pore separation in the tungsten plug of the L-cathode, and it is concluded that the whole of the exposed tungsten surface will contribute to the emission. There is a brief theoretical discussion of the relation between the lifetime of an adsorbed atom and the time taken for a surface over which W atoms are spreading to reach a stable level of emission. Experiments on the activation of two W wires, one well cleaned and the other heat-treated like an L-cathode, suggested that the emitting surface consists of a nearly complete oxygen monolayer followed by a complete barium monolayer, this surface giving lower work function and longer sticking time of Ba than clean W. It is concluded that the emission actually obtained is close to the maximum that can be expected from a dispenser cathode of this type.

Article by E. S. Rittner R. H. Ahlert & W. C. Rutledge *J. Appl. Phys.* **28**, Feb. 1957 156-166

59/IV

Modern Thermionic Cathodes

United Kingdom. The author reviews the developments in cathodes since the second world war during which time the stringent requirements of microwave valves have shown the oxide cathode to be insufficient in several respects. Choice of cathode material is governed by three factors, melting point, ease of fabrication and work function. Three groups of cathodes were considered (*a*) pure metals, (*b*) atomic films, (*c*) oxide emitters. The author summarises the present position by stating that the oxide cathode is still the most widely used cathode and that all other types have somewhat specific applications. In general the L-type and impregnated cathodes require much more power and will be used in the more expensive valves where the oxide cathode fails and heater wattage is not a prime consideration. Operating conditions of the cathodes with expected life are contained in a table.

Article by R. W. Fane *Wireless World* **63**, Oct. 1957 488-490

60/IV

The Emission from Oxide Cathodes in Low Pressure Discharge

United Kingdom. It is shown that measurements made in vacuum on oxide-coated discharge cathodes are unreliable as a measure of the properties of the cathode in the gas atmosphere of the discharge. Several known methods of measuring the zero-field emission of cathodes in gas atmospheres are found to give rise to difficulties when applied to oxide cathodes in several millimetres pressure of rare gas. A method is described in which these difficulties are avoided and it is applied to a study of cathodes of the fluorescent lamp type. It is found that the zero field emission of these cathodes at operating temperatures is considerably higher than the thermionic emission measured in vacuum, but still much smaller than the discharge current which they normally pass. It is shown that the various secondary and field emission processes play a prominent part in the normal operation of these cathodes which, in fact, need not necessarily be particularly good thermionic emitters. (*Author*)

Article by M. A. Cayless *Brit. J. Appl. Phys.* **8**, August 1957 331-336

61/IV

The Effect of Air Exposure on Various Cathodes for Demountable Vacuum Systems

United States. Electron emitters have been investigated by the authors in order to determine the best type for use in demountable vacuum systems. Those investigated include both nickel and tungsten matrix cathodes and the more common oxide-coated nickel cathode. The tests were carried out in a coplanar diode system using a water-cooled copper anode. Tests were carried out measuring emission variations with time and number of exposures to the atmosphere. During each exposure, lasting one hour, the anode was chemically cleaned and hydrogen fired. On each evacuation to 5×10^{-6} mm. mercury the cathodes were first activated by normal methods and then a d.c. anode voltage of 100 volts applied and the emission measured as a function of time. Three types of nickel matrix cathodes were investigated, firstly the 'moulded' nickel matrix, secondly the impregnated nickel matrix and thirdly two types of nickel matrix in which the active material was dispersed throughout the body of the cathode. In all three cases and also in the case of a simple oxide-coated cathode the emission decreased with increasing numbers of exposures. This is attributed principally to flaking of the coating which was observed to be worse during humid weather. The diffusion nature of the matrix cathodes which it was thought would give beneficial results was overshadowed by this flaking phenomenon. Two types of barium tungsten matrix cathodes were investigated, namely the 'impregnated' and 'pressed' varieties from Philips Laboratories. In both of these the emission changes with increasing exposures were less marked provided the anode was cleaned on each exposure, but the activation times after each exposure became progressively longer. The authors reach the conclusion that the nickel matrix cathodes are not true diffusion cathodes whereas the tungsten matrix cathodes are. In general if the long re-activation times can be accommodated the tungsten cathodes are better, but due to self-poisoning effects the anodes must be thoroughly cleaned during each exposure. The simple oxide-coated cathode is perhaps the best although it involves control of humidity. The authors are proceeding with experiments to control the flaking of oxide cathodes with a view to making them more useful for demountable vacuum systems.

Report by G. A. Hass & J. T. Jenson, Jr. *U.S. Naval Research Lab. Rep.* No. 4856 November 1956

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Use of Oxide Cathodes in Demountable Vacuum Systems

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(Reference only)

Article by G. A. Hass & J. T. Jensen Jr. *Rev. Sci. Instrum.* **28**, December 1957 1007-1010

Some Problems on Disc-Sealed Planar Triodes

63/IV

Japan. Problems associated with the use of higher temperature oxide cathodes in ultra high frequency disc-sealed triodes are discussed. Evaporation of the coating material on to the grid is such that the build up of evaporated material causes changes in the grid cathode spacing and also grid mesh dimensions with their resultant changes in value characteristics. The theory of the build up is presented together with some examples of the order of magnitude of change in anode current and amplification factor with time using different values of cathode temperature. From these it can be seen that cathode temperature is very critical. Sputtering on the glass bulb, between the grid and cathode due to cathode evaporation, affects the interelectrode capacitances in such a way as to de-time the oscillator if sputtering becomes pronounced. The grid-cathode capacitance increases rapidly for the first 200 hours of life thereafter settling down as saturation occurs. Graphite was coated on the bulb in some test valves in order to prove that the sputtered film was the real cause of the changes. On admitting air to a heavily sputtered valve the sputtered film gradually turned white in a way which suggested that barium was the main constituent. Gases released from the electrodes during life are of two varieties, firstly those released by thermal effects and secondly those released by dissociation. The former can be eliminated by pre-treatment of component parts and heating during evacuation. The latter, as the authors show, is only removed by electron bombardment of the electrode concerned. The chief component produced by dissociation is oxygen from slight oxide layers and is particularly bad in its effect on the cathode due to its high activity. In order to minimise this effect, during processing the electrodes must be electron bombarded to a higher energy level than is experienced during normal running. Evidence is presented showing that tubes thus treated are better than those receiving normal radio valve processing schedules. Frequency drift is clearly related to cathode activity. The authors attribute frequency drift to poor initial emission which leads to a slow build up of back bombardment with its resultant condition causing frequency drift. Factors affecting tube life are considered. Increases of amplification factor limit life. This has been attributed to changes in electrode spacing during life which has two results, namely, it causes lowering of anode current and also spatial irregularity of μ in a direction parallel to the cathode surface. In conclusion the authors suggest that it is necessary to design a tube for ultra-high frequency work with as low a cathode temperature as possible and using cathode material with a low evaporation rate.

Article by H. Nishio T. Nemoto & H. Murakami *Le Vide* **67**, Jan-Feb. 1957 9-19

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Production of Television Tubes

64/IV

See Abstract No.: 77/I

The Suppression of Screen Grid Emission by Carbon

65/IV

United Kingdom. The performance of vacuum tubes may be degraded by thermionic emission from the grids. It is caused by barium and barium oxide evaporated from the cathode during manufacture, and suppression of the effect is relatively simple when the grid is operated at a low temperature. Screen grids, however, may be operated at relatively high power. Carbon has a high emissivity so that it would reduce the grid temperature (and therefore the thermionic emission) at a given power level. In fact, greater suppression of emission is observed than can be accounted for by this effect, and experiments are described which lead to an explanation. It is shown in a control experiment that pure tungsten and molybdenum behave similarly with regard to thermionic effects when coated with barium and with barium oxide. In the main experiment a tungsten wire is used as a standard for comparison, and a strip of molybdenum coated with carbon is used to observe the effects of carbon. Emission from both tungsten and carbon-coated molybdenum was too low to account for the observed values in actual valves, when coated with pure barium. When coated with barium oxide, emission from the tungsten at 1,000°C at first rose, and then slowly fell, while the carbon-coated molybdenum showed a very rapid fall over the first 10–20 minutes. Further experiments with the carbon-coated molybdenum showed that the initial rapid fall was very largely dependent on temperature. At about 900°C very little change was observed, while below this temperature a rise was measured. A subsidiary experiment showed that the substance evaporated from a mixture of carbon and barium carbonate is barium, not barium oxide. The conclusion is that at high temperatures carbon reduces the thermionic emission from barium oxide by causing evaporation of barium as a result of the reaction, $BaO + C = Ba + CO$, and this is confirmed by a graph of emission against time of a double logarithmic scale, showing an abrupt change of slope at the point where all the oxide has been converted to the metal. The apparatus for the experiments consisted of tungsten and molybdenum (or carbon-coated molybdenum) wires or strips mounted together with suitable barium or barium oxide emitters inside a nickel anode *in vacuo*. The barium was obtained from a 'batalum' getter deposited on a molybdenum strip. A spiral of platinum wire sprayed with barium carbonate suspension provided a source of barium oxide.

Article by J. A. Champion *Brit. J. Appl. Phys.* **7**, Nov. 1956 395-399

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The Life and Reliability of Valves

Holland. Valve failure rate is defined as the percentage of failures per unit time. For any batch of valves it is normally high for a short initial period, constant for a long period, and then high again. The life of the valve is then defined as the time required to reach the onset of the second period of high failure rate; and the reliability is the reciprocal of the failure rate in the constant region. Failures may be sudden, as in the case of a short circuit for example, or gradual, as in the case of a slow fall-off of gain. These failures may be reduced by careful manufacture and by careful use. The cathode depends for its performance on the presence of a small amount of free barium in its coating, and sub-standard performance results from the removal of the barium by residual gas ions or by substances released from the material of the valve under electron bombardment. Good degassing, scrupulous cleanliness, and shielding from electron bombardment help with these problems. Evaporation from the cathode causes poor insulation within the valve, and grid emission may also result. Broken connections can be overcome by using carefully controlled welding techniques, while short-circuits can be avoided by removal of foreign particles (dust during assembly, flakes from mica spacers). The risk of heater-cathode short circuits can be reduced by providing a long insulating path between heater and cathode, or by coating both heater and cathode with alumina. Valve-to-holder contacts are improved by gold plating the pins. Characteristic spreads are reduced by a short ageing period, and by using more rigid structures. Shock and vibration resistance is improved by allowing no free play in any part of the valve, using light components and raising the frequency of mechanical resonances. By methods such as these a failure rate of 0.5% per 1,000 hours and a life of 10,000 hours have been achieved.

Article by K. Rodenhuis H. Santing & H. J. M. van Tol *Philips Tech. Rev.* **18**, Dec. 1956 181-192

67/IV

Triodes and Tetrodes for U.H.F.–S.H.F. Operation

Great Britain. A review of advances made in the last 10 years in 'conventional' valves for frequencies above 300 Mc/s. The authors deal mainly with manufacturing technology. Glass as an envelope material has been advantageously replaced in some cases by a ceramic material, allowing the temperature of baking on the pumps to be as high as 750°C (cf. 550°C for hard glass) which gives a better vacuum and allows a higher anode temperature in operation. The temperature of baking is then limited mainly by the vapour pressure of the brazing metal. A number of new alloys, having good wetting properties and low vapour pressure are mentioned. Close dimensional tolerances can be achieved in brazed assemblies of metal and ceramic parts, and the ceramic parts have good thermal shock resistance and low radio-frequency losses. There is some discussion of cathode materials, the importance of very high emission current densities for pulse operation at these frequencies being pointed out. The Lemmens 'dispenser' cathode is mentioned and the recent development of sheet thoriated tungsten, both of which permit the use of higher steady potentials, and hence larger spacings. Grids are made from gold-plated tungsten, or platinum-clad molybdenum wire, the coatings being to absorb Ba and Th respectively, which otherwise deposit during operation and cause thermionic emission from the grid. Grid wires as fine as 5 micron, with as many as 1,000 turns/inch have been used. Several grid fabrication methods are referred to. There are brief notes on noise factors and on the types of circuit used and two tables giving summarised information on the dimensions, operating conditions and performance of a selection of modern microwave valves. Twenty-nine references are appended.

Article by C. A. Tremlett & A. D. Williams *Electronic Engng.* **30**, May 1958 335-340

68/IV

A New Cold-Cathode Trigger Tube I

Great Britain. This article, in three parts, is mainly concerned with the characteristics and applications of a new type of close-tolerance trigger tube. In Part I, which is abstracted here, are considered the general principles of operation of trigger tubes, factors influencing their reliability and the construction and processing of the new tube, the type Z803U. The operation is contrasted with that of a hot-cathode tube; cumulative ionisation and ion bombardment of the cathode are necessary to give a self-maintaining discharge, so that the operating voltage is of the order of 100V. Also, the trigger electrode, unlike the thyratron grid, operates by establishing a subsidiary glow discharge to the cathode which then transfers to the anode. The input signal is thus not merely a bias, but must supply a small quantity of energy. If the source of signal has a high impedance, it may nevertheless charge a capacitor which then discharges through the trigger/cathode gap when its p.d. is high enough. The mechanism of the glow discharge is briefly discussed; the trigger tube operates ordinarily in the region of 'normal glow', though under pulse conditions the current may enter the 'abnormal glow' region. The possibility of initiating the trigger discharge at a moderate voltage is considered; the necessary primary electron current may be obtained by photo-emission, stray ionisation from an auxiliary priming discharge or from a radioactive source. In this tube, a pure molybdenum cathode is used, for good life and stability; photo-emission is therefore small, and for reliability it is considered inadvisable to depend on ambient illumination. A priming discharge is therefore used, being maintained throughout the operation of the tube. A very small quantity of uranium oxide, painted on the inside of the envelope, causes enough ionisation to ensure easy starting of the priming discharge. The factors governing the ionisation time are briefly discussed; this depends mainly on the 'over-voltage' applied to the trigger and can vary between 20 and 2,000 microsec for this tube. The deionisation or recovery time is also considered; this is 2–3 millisec. Another effect mentioned is 'trigger hysteresis'—a variation in the voltage necessary to start the trigger discharge, during the first 100 millisec after extinction. The construction of the tube is described, with a clear sketch showing the electrode assembly. There are no micas, all electrodes being mounted directly on the glass button base. The cathode is a U-shaped plate of pure molybdenum, mounted vertically and supported on three pins for rigidity. The priming anode is a small wire electrode, mounted close to the inside of the U, whilst the trigger electrode is another wire mounted close to the outside. The anode is a nickel wire, mounted vertically at the opposite side of the tube and partially shielded by a glass tube. During processing, the tube is operated long enough to sputter a layer of metal from the surface of the cathode on to the glass envelope. This serves to give a clean, stable cathode surface, to clean-up residual gas and prevent gas from the glass entering the tube, and to shield the assembly from ambient light. Graphs show the variation in critical trigger voltage during 4,000 hr. shelf life and 10,000 hr. running.

Article by K. F. Gimson & G. O. Crowther *Electronic Engng.* **29**, Oct. 1957 462-468

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A Cold-Cathode Trigger Tube

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Great Britain. An article discussing the design and manufacture of a low-cost trigger tube—the type GTR120W. The mechanism of the cold-cathode discharge is first briefly discussed: A minute initial current is necessary to provide a primary source of electrons. This current can be obtained by photo-emission from the cathode or by the use of radioactive materials or a priming discharge. For cheapness, this tube makes use of photo-emission—the cathode being a nickel wire which is coated with a layer of BaO and Ba by the firing of the getter. The tube has to be operated in ambient illumination of at least 5 foot-candles. To reduce damage of the coated cathode surface by ion bombardment, a pure gas of low atomic weight is used. The manufacture of the tube is largely mechanised. It has three electrodes, each consisting of a composite wire, a nickel section acting as the electrode and a borated-copper sealing section. The getter ring is welded to the end of the cathode wire, and the three wires are mounted in a glass bead. After adjusting the critical cathode-trigger spacing, the wires are sealed into a 'pinch' and then into the envelope. The tubes are pumped on an automatic rotary pumping table, being leak-tested automatically at the beginning of the schedule and again after baking. No details are given of the pumping system, though it is remarked that it is necessary for some residual oxygen to remain to give BaO on firing the getter. After filling and gettering, tubes pass to an automatic rotary ageing and testing machine. At any testing position, rejected tubes are removed and dropped into a box for inspection. Tube characteristics are discussed; the anode is initially made about 300V positive with respect to the cathode. The tube does not, however, fire until the trigger electrode, which is much closer to the cathode, is made about 170V positive. The anode takes over the discharge from the trigger and a larger current passes; the discharge to the trigger electrode is then extinguished by a rise in the cathode potential due to the flow of the larger current through a resistor in the cathode lead. The maintaining voltage is about 105V; jumps of up to 10V may occur, but are not necessarily serious in switching service. Suitable operating conditions are briefly discussed, and a circuit is given for coupling one tube to another.

Article by A. Turner *Electronic Engng.* **30** April 1958 166-169

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Thermionic Constants and Sorption Properties of Hafnium

70/IV

United States. Hafnium was chosen for these experiments since it was hoped that its high melting point and low work function would make it suitable for use in a system designed to measure the ejection of electrons by ions of the noble gases. The work to date indicates that the sorption properties of the metal are such that the work function often becomes high and that this change is irreversible. Thermionic emission experiments on hafnium at temperatures between 1,250°K and 1,820°K yielded a work function of 3.60 eV and a thermionic constant 'A' of 22.9 A/cm²/°K². Measurements were made on two samples of hafnium metal each mounted in a sample vacuum system consisting of a glass bulb and an ionisation gauge connected to a two-stage mercury diffusion pump through a liquid nitrogen trap. Vacuum treatment of the samples was as follows: After baking at 400°C for one day, the metal was heated for 15 hours at temperatures between 2,000° and 2,100°K. During this time the pressure rose from 10^{-8} to 5×10^{-8} mm. Hg. After cooling, the residual pressure was about 1×10^{-9} mm. Hg. The work function was then obtained from the slope of the Richardson Law graph of $\frac{i}{T^2}$ against $\frac{1}{T}$. Temperatures were measured by a micro-optical pyrometer and a red filter was used. Air was then admitted to the system to permit minor modifications and the apparatus was then evacuated and baked at 400°C. The ribbon of hafnium was then heated intermittently to 2,150°K for a total hot time of 46 hrs. the pressure being below 6×10^{-10} mm. Hg at all times throughout the heating. After 15 hrs. heating, the sample was kept cold for 64 hrs.; the pressure was 2.5×10^{-10} mm. Hg. The temperature was then raised to 2,150°K in 15 seconds and the pressure rose from 5×10^{-10} to 7×10^{-10} mm. Hg. and returned to 6×10^{-10} mm. Hg with the ribbon still hot. This showed that no appreciable desorption was taking place even after 64 hrs. in the cold state. The second sample of hafnium was used as a target in an apparatus for studying the electron ejection by ions. It was more brittle than the first sample and more contaminated with gas. Its work function was found to be 3.91 eV and the constant 'A' to be 20.5 A/cm²/°K² after repeated flashing at 2,150°K. The vacuum treatment was followed as for the first sample. The electron ejection experiments indicate that the surface of this sample was covered with an appreciable fraction of a monolayer immediately after cooling from high temperature. The author concludes that the work function of hafnium increases as the metal irreversibly absorbs gas on heating.

Article by H. D. Hagstrum *J. Appl. Phys.* **28**, March 1957 323-328

Work-Function Studies of Germanium Crystals Cleaned by Ion Bombardment

71/IV

United States. In these experiments, the work function of a gold-plated reference surface was measured directly by the Fowler photo-electric method, and that of the germanium by using the Kelvin method to obtain the c.p.d. between the Ge and Au. Either the reference surface or the Ge crystal could be advanced to the centre of a spherical vacuum chamber, where a beam of u.v. light could be directed on to them from a monochromator. When c.p.d. measurements were to be made, the reference surface was placed at the centre of the chamber and the Ge advanced to within a few mm. of it, the two being connected in series in a circuit containing the input resistance of a d.c. amplifier and a potentiometer. The potentiometer output was adjusted until no amplifier output was obtained on moving the reference relative to the Ge. The crystal was mounted in contact with graphite on the end of a quartz tube. When the crystal was to be treated, it was retracted into a side-arm of the vacuum chamber, where it could be heated by electron bombardment from shielded filaments, or ion bombarded in Ar, electron emission from the filaments serving to ionise the Ar and so allow operation at a lower pressure, with the idea of diminishing back diffusion of sputtered material on to the crystal. The main tube was baked for 5 hrs. at 350–400°C, after which an ion gauge reading of 5×10^{-10} mm. was obtained, with a liquid N_2 trap in use. The residual partial pressure of O_2 was estimated to be no more than

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2×10^{-11} mm. The crystals used had a volume of about 1 c.c., a surface of about 1 cm² and were oriented to within $\frac{1}{4}°$. They were etched in 'CP–4' (acetic, nitric, hydrofluoric acids and bromine) and rinsed in HF and water. The usual cleaning procedure in the vacuum chamber was to repeat cycles of 20 hr. heating at 650°C, 10 min. of ion bombardment and 15 min. of annealing at 500°C until the total time of heating was about 150 hr. and ion bombardment time of 90 min. The bombardment was carried out at 400–500 V, 60–100 uA/cm² and an argon pressure of 5×10^{-4} to 3×10^{-3} mm. Hg. There was evidence that Ar was driven into the crystal during bombardment and then liberated during annealing, but microscopic and electron-diffraction examination showed no signs of preferential etching of a type previously reported. Values of work function of 4.78 and 4.73 eV were obtained for resistivities of about 40 and less than 1 ohm-cm, respectively, the values being very insensitive to variations in Fermi level due to varying impurity concentration. Possible explanations of this are discussed. There was little evidence of differences more than about 0.2 eV between different crystal faces. There is an extensive account of the changes in work function due to exposure to oxygen and other gases at low pressures.

Article by J. A. Dillon Jr. & H. E. Farnsworth *J. Appl. Phys.* **28**, Feb. 1957 174-184

72/IV

A Scintillation Counter for Soft-β Paper Chromatograms

(Reference only)

Article by K. Steenberg & A. A. Benson *Nucleonics* **14**, Dec. 1956 40-43

73/IV

Photo-Ionisation in Self-Quenching Geiger Counters

Pakistan. It is established that the ultra-violet radiations produced from excited argon atoms in a Townsend avalanche occurring in a self-quenching Geiger counter are energetic enough to give rise to photo-ionisation of the polyatomic gas of the filling mixture. The experimental procedure is described fully.

Letter by R. M. Chaudri M. Y. Khan A. Khawaja & R. Dar *Nature* **181** 1526-1527

74/IV

Low Flow Variable Leak

See Abstract No.: 127/II

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Glass-to-Metal Seals

United States. The article states the conditions which must be satisfied by glass-to-metal seals. Five types of seals are then described. Expansion metal seals are used where the linear coefficient of expansion of the metal and glass are nearly identical. The importance of an oxide layer on the metal on the bonding is stressed. A table of suitable glass-metal combinations is given. Housekeeper-type seals permit sealing metal and glass tubes of different expansion coefficients but the metal must be sufficiently thin to allow it to flex under stress without failure of the seal. Graded seals are short sections of glass tubing made up of thin layers, each of progressively lower expansion coefficient. A new type of seal the 'internal seal' is described. This is intended for use where conditions do not permit either an expansion metal or a Housekeeper seal. The fifth type of seal is the metallised glass seal; reference is made to the use of this technique to produce precision inductances and printed circuits. The various types of seals are illustrated by photographs of typical examples.

Article by T. H. Mariner *Prod. Engng.* *Jan.* 1957 188-192

76/IV

Sealing Glass to Metals

Germany. The technique of fusing Pt, Cu, Mo, W and Cr–Fe wires to glass is described in detail. Several illustrations showing the right and wrong method of making glass-metal seals are given, high-lighting the need to make the glass flow along the wire on both ends of the seal. The cleaning processes for the wires are discussed specifying *inter alia* 20% H_2SO_4 solution etching for Cr–Fe wires or 10% NaOH solution etching for W wires with subsequent rinsing with detergents. The need for controlling the oxygen content of the flame during fusing is stressed repeatedly. Useful details about the colour changes of the wires during processing are included.

Article by R. Kleinteich *Glas-Email-Keramo-Technik* **9**, September 1956 355-357

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Simple Refractory-Metal and Glass-Metal Seals

(Reference only)

Note by W. D. Jameson *J. Sci. Instrum.* **35**, February 1958 73

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Method for Making Vacuum Feedthrough Terminals

78/IV

South Africa. A Standard Kovar/glass skirted seal has been used to make an insulated electrical feed through into a vacuum system. The vacuum seal is effected by screwing down the skirt of the seal on to a soft aluminium foil gasket, by means of a specially shaped ring, designed to avoid cracking the glass.

Note by
J. W. L. de Villiers
Rev. Sci. Instrum
29, June 1958
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